NOVA

User manual

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1 – Introduction

NOVA is designed to control all the Autolab potentiostat/galvanostat instruments with a USB connection. It is the successor of the GPES/FRA software and integrates two decades of user experience and the latest .NET software technology.

NOVA brings more power and more flexibility to the Autolab instrument, without any hardware upgrade.

NOVA is designed to answer the demands of both experienced electrochemists and newcomers alike. Setting up an experiment, measuring data and performing data analysis to produce publication ready graphs can be done in a few mouse clicks.

NOVA is different from other electrochemical software packages. As all electrochemical experiments are different and unique, NOVA provides an innovative and dynamic working environment, capable of adapting itself to fit your experimental requirements.

The design of NOVA is based on the latest object-oriented software architecture. NOVA is designed to give the user total control of the experimental procedure and a complete flexibility in the setup of the experiment.

This user manual provides in-depth details about the functionality of NOVA. It explains the most important features of the software, using many practical examples. The user manual has four chapters:

- Chapter 1 provides an overview of NOVA
- Chapter 2 describes the procedure building process
- Chapter 3 describes the measurements with NOVA
- Chapter 4 provides information regarding the data analysis

1.1 – The philosophy of NOVA

NOVA differs from most software packages for electrochemistry.

The classic approach used in existing electrochemical applications is to code a number of so-called *Use cases* or *Electrochemical methods* in the software. The advantage of this approach is that it provides a specific solution for well defined experimental conditions. The disadvantage is that it is not possible to deviate from the methods provided in the software. Moreover, it is not possible to integrate all the possible electrochemical methods, since new experimental protocols are developed on a daily basis. This means that this type of software will require periodical updates and will necessitate significant maintenance efforts.

Figure 1.1 shows a typical overview of a classic, *method-based* application for electrochemistry.

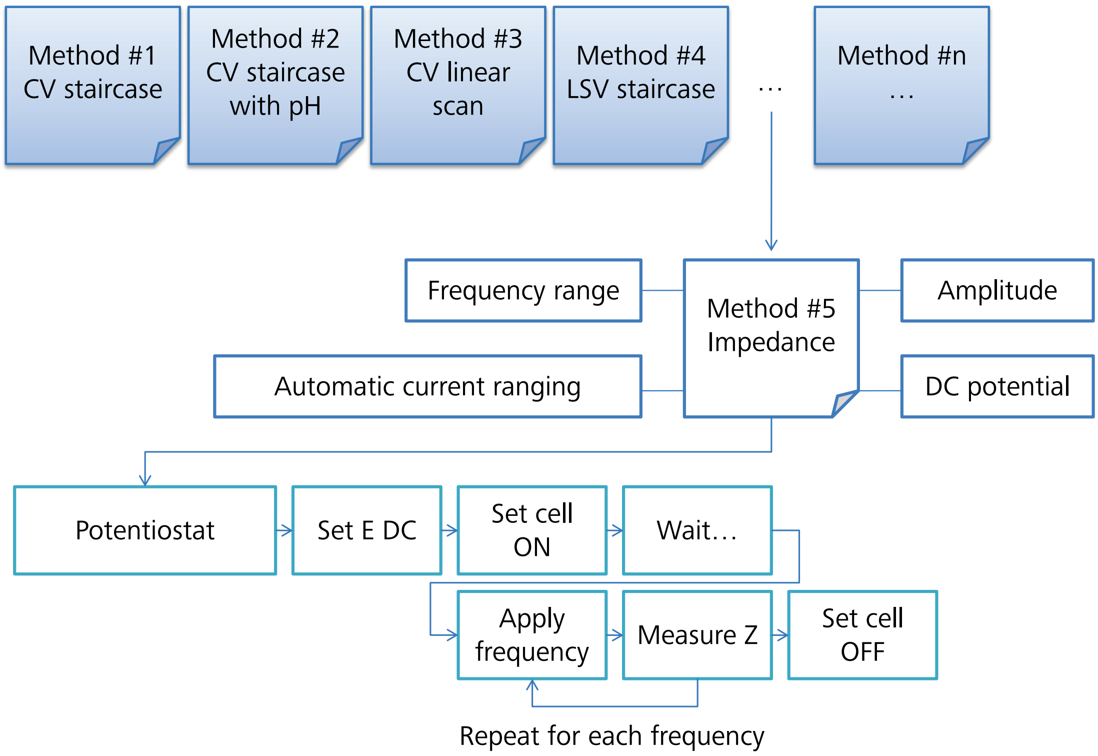


Figure 1.1 – Schematic overview of a method-based software

In a method-based application, the user chooses one of the *n* available methods and defines the available parameters for the method. When the measurement starts, the whole method is uploaded to the instrument where it is decomposed into individual, low-level instructions. These are then executed sequentially until the measurement is finished.

If the method required by the user is not available, the user will have to wait until the method is implemented in a future release.

NOVA has been designed with a completely different philosophy. Rather than implementing well defined methods in the software, NOVA provides the users with a number of basic **Objects** corresponding to the low-level functions of the electrochemical instrument. These objects can be used as **building blocks** and can be combined with one another according to the requirements of the user in order to create a complete experimental method. In essence, the scientist uses NOVA as a programming language for electrochemistry, building simple or complex procedures out of individual commands. The instructions can be combined in any way the user sees fit. Rather than providing specific electrochemical methods to the user, NOVA uses a **generic approach**, in which, in principle, any method or any task can be constructed using the available commands.

Figure 1.2 shows the NOVA strategy, schematically.

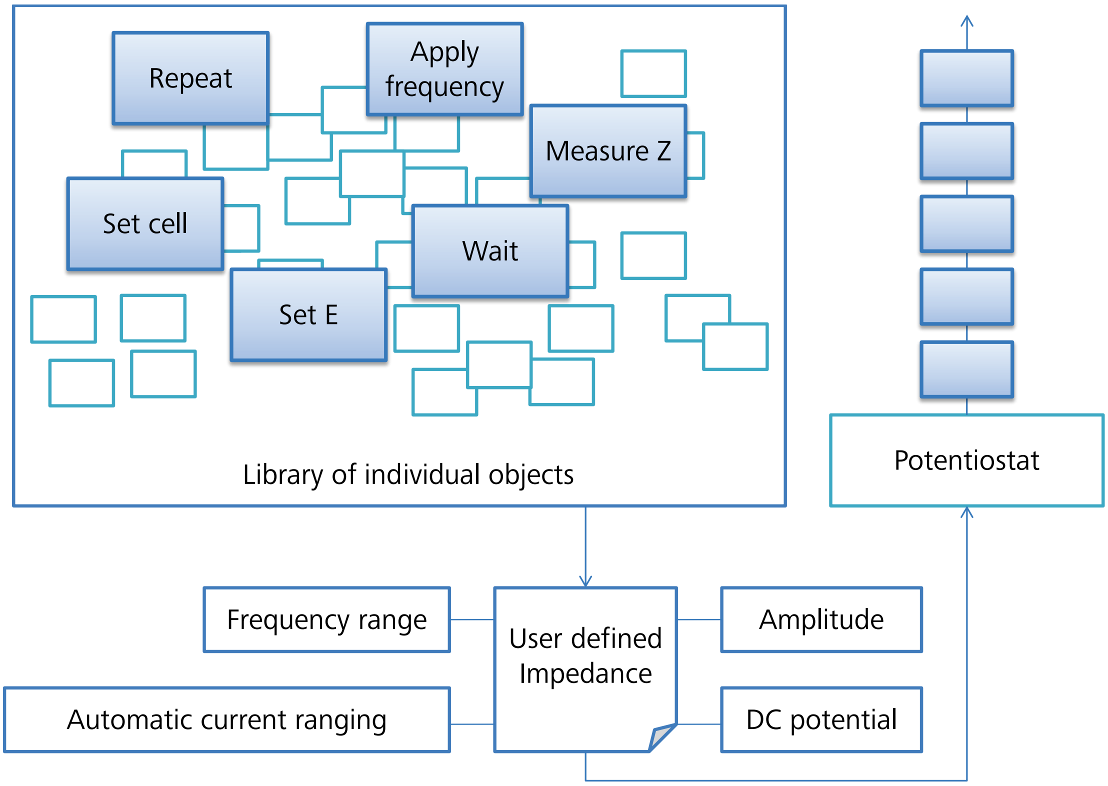


Figure 1.2 – Schematic overview of the object-based design of NOVA

The NOVA approach allows the user to program an electrochemical method in the same *language* used by the instrument.

This new object-based design philosophy has led to the current version of NOVA. As any task can be solved generically, the software is slightly less intuitive than a method-based application. Depending on the complexity of the experiments, the learning curve can be more or less long. For this reason, we advise you to study carefully this User manual as well as the Getting started.

Because of the large number of possibilities provided by this application, it is not possible to include the information required to solve each individual use case. A number of typical situations are explained using stand-alone tutorials (refer to the Help menu – Tutorials). These tutorials provide practical examples.

In case of missing information, do not hesitate to contact Metrohm Autolab at the dedicated nova@metrohm-autolab.com email address.

1.2 – Working with NOVA

This section briefly describes some features of the NOVA user interface. More details will be provided in the next chapters on procedure setup, data measurement and data analysis.

1.2.1 – The toolbar buttons

Clicking a specific button in the toolbar can perform most of the NOVA operations. Figure 1.3 shows an overview of the NOVA toolbar and its buttons.

Figure 1.3 – The NOVA toolbar

The toolbar shows a number of buttons, some of which might be grayed out (in which case the attached instruction cannot be performed) or highlighted (which indicates a persistent state of NOVA).

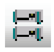
This section provides an overview of the toolbar buttons.

 **New Procedure**, clears the procedure editor frame

 **Save Procedure**, saves the currently edited procedure in the database

 **Print Procedure**, prints the currently edited procedure on the default printer

 **Setup view**, switches to the Setup view (highlighted button, i.e. active status)

 **Multi Autolab view**, switches to the Multi Autolab view

 **Measurement view**, switches to the Measurement view

 **Analysis view**, switches to the Analysis view

 **Start measurement**[1], starts the measurement by executing the currently edited procedure

 **Pause measurement**[2], pauses the measurement (click the start button again to continue)

 **Skip command**[2], interrupts the current command and jumps to the next command in the procedure

[1] This button is inactive during the measurement.
[2] This button is active during the measurement.

 **Abort measurement**[2], stops the measurement

 **Show/Hide Autolab display**[3], shows or hides the Autolab display

 **Show single plot during measurement**, is used to display a single plot in the measurement view

 **Show two plots vertically tiled during measurement**, is used to display two plots, vertically tiled in the measurement view

 **Show two plots vertically tiled during measurement**, is used to display two plots, horizontally tiled in the measurement view

 **Show four plots during measurement**, is used to display four plots in the measurement view

 **Link parameters**[4], is used to link two different parameters

 **Unlink parameters**[4], is used to break an existing link between two different parameters

 **Collapse one level**[4], used to contract the displayed procedure into a more compact view (opposite of Expand one level)

 **Expand one level**[4], used to expand the displayed procedure into a more detailed view (opposite of Collapse one level)

 **Show all links**[4], expands the currently edited procedure in order to show all existing links

 **Undo**[5], cancels the last performed action, if possible

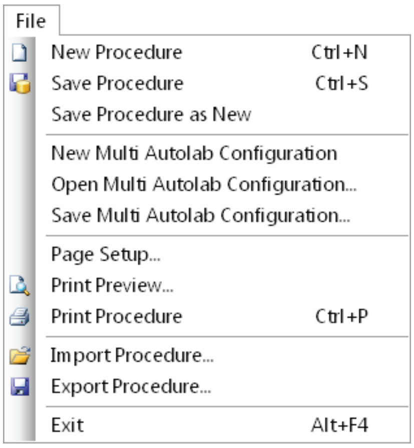
 **Redo**[5], repeats the last cancelled action, if possible

 **Clear measurement plot**[6], clears the contents of all plots in the measurement view, or a specific plot (1 to 4)

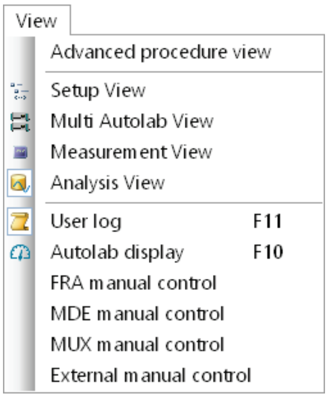
[3] The Autolab display can also be shown by pressing the F10 key.
[4] Only active in the Setup view. This button is greyed out in the two other views.
[5] The following actions can be undone/redone: delete command, move command, insert command, link/unlink command parameter.
[6] Only active in the Measurement view. This button is grayed out in the two other views.

1.2.2 – The toolbar menus

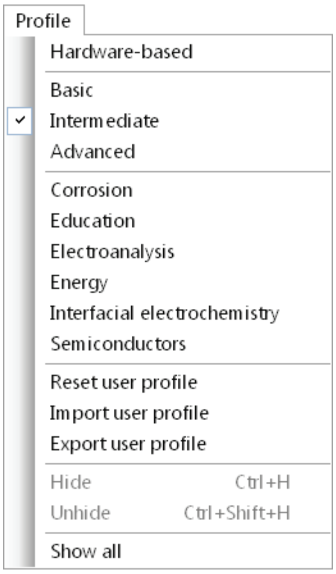
Some NOVA operations require the use of instructions located in the drop down menus of the toolbar. A short overview of the menus is given below.



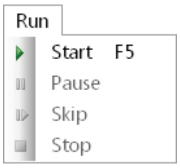
File menu, shows all the instructions related to creating, saving and printing a procedure. Multi Autolab related items are also found in this menu. Some of the instructions have a corresponding button that can be present in the toolbar and a convenient keyboard shortcut.



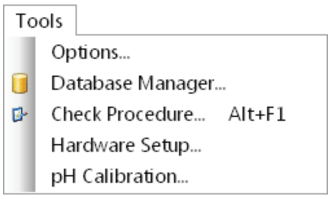
View menu, shows all the view-related commands. It also provides access to the control of some of the accessories. Some of the instructions are highlighted (indicating an active status).



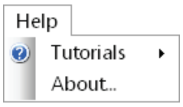
Profile menu, shows all the available profile schemes in NOVA. The profiles are arranged in three groups: Hardware based, Level based (basic, intermediate and advanced) and Application based. More information on the use of profiles is provided in Section 1.6.1. Some of the profiles are highlighted (indicating an active status).



Run menu, shows the Start, Pause, Skip and Stop instructions. The pause, skip and stop instructions are only active during the measurement.



Tools menu, gives access to the options, the database manager, the procedure check and the hardware setup.



Help menu, used to access the Tutorials[7] .

1.2.3 – Views and frames

The NOVA user interface uses four different **views.** Three of these are dedicated to a specific part of the experiment. A fourth view is dedicated to the control of multiple Autolab instruments[8].

Procedure setup, measurement and data analysis are performed sequentially. Each part of the experiment is carried out in a specific view of NOVA (see Figure 1.4).

[7] The tutorials require Acrobat Reader to be installed.
[8] More information on the use of the Multi Autolab view can be found in the Multi Autolab tutorial, available from the Help – Tutorials menu.

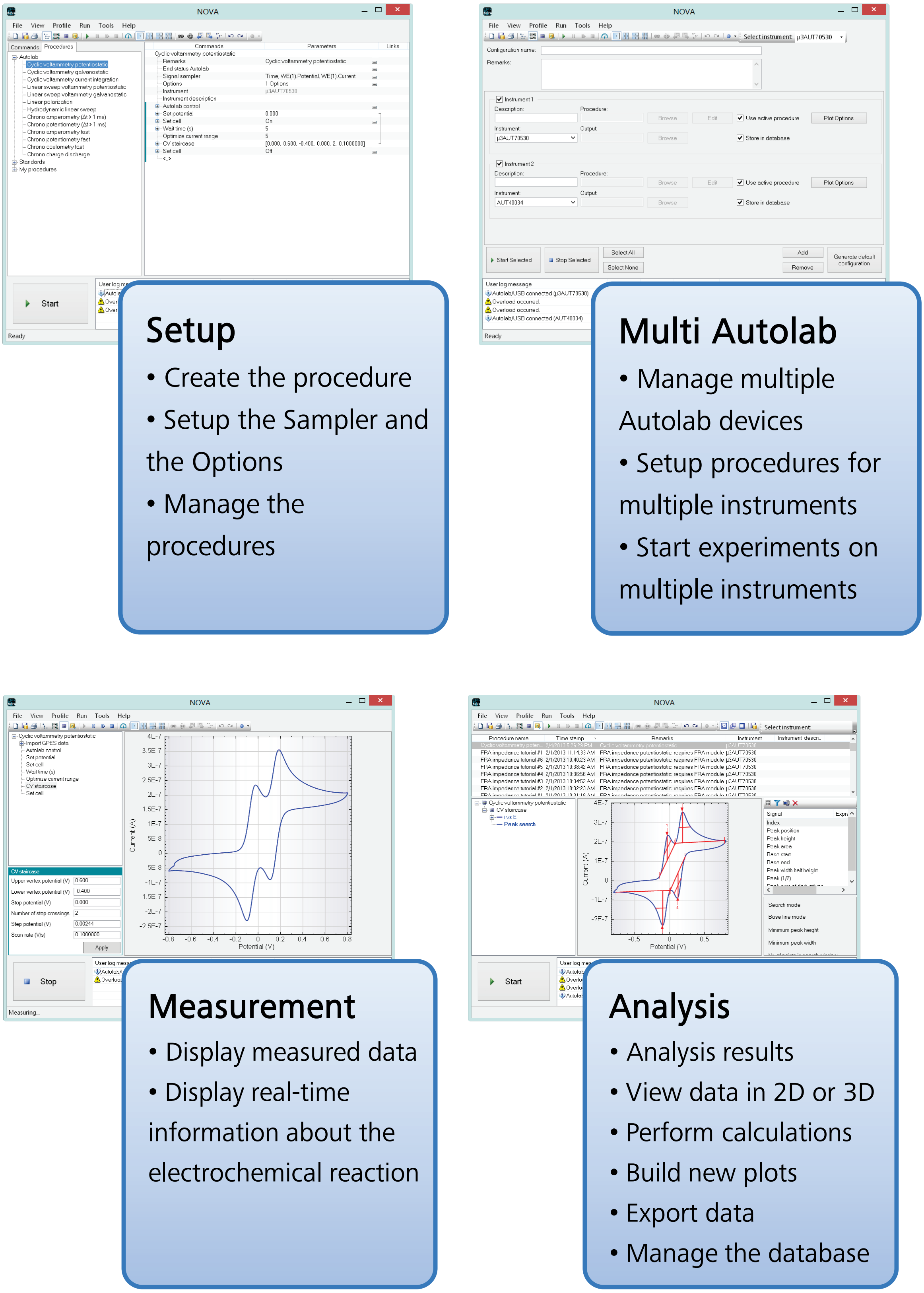


Figure 1.4 – The four views of NOVA

The **Setup view** provides a framework for procedure editing. It is used to create procedures, setup the way the data points are gathered and to manage user-defined procedures through the use of a custom made database. This part of NOVA is described in more detail in **Chapter 2** of this user manual.

The **Measurement view** displays real time data points during an ongoing measurement. It also provides an overview of the progress of the procedure. **Chapter 3** describes the Measurement view.

The **Analysis view** is used to perform data analysis on previously recorded measurements. It lets the users plot experimental data in a 2D plot or in a 3D plot. This view is also used to generate new plots and to perform calculations on the measured data using a built-in spreadsheet. The Analysis view also provides an overview of all the performed measurements and the user-defined results database. This view of the software is described in extensive detail in **Chapter 4** of this manual.

The **Multi Autolab view** is used to configure multiple instruments connected to the same computer or Multi Autolab instruments fitted with multiple channels and to program the measurements to be performed on these devices. The use of the Multi Autolab view falls outside of the scope of this User manual and more information can be found in the **Multi Autolab tutorial**, available from the Help menu.

Note

The Multi Autolab view is only used to configure multiple instruments connected to the same computer. Procedure setup, experiment monitoring and data analysis is still carried out in the respective views of the software.

Note

Since the different parts of an experiment are performed in different views of the software, Nova lets users perform data analysis in the Analysis view *while* a measurement is running in the Measurement view. Analyzing data does not interfere with the measurement itself.

1.3 – NOVA workflow

This section provides some details about basic concepts related to the way NOVA works. More information is provided in the rest of this document and in the NOVA Getting started manual.

1.3.1 – The commands

NOVA works with individual commands. Each command can be used to perform a specific action like defining a potential value, or performing a cyclic voltammetry measurement. The following nomenclature is used to describe commands in NOVA:

- **Command:** this is the name of the command, by which it is identified in the software. The name of the command also indicates the role of the command in the procedure.
- **Command parameter:** this is a parameter of a given command. A single command can have one or more command parameters. Each command parameter specifies a value required for the Command to work as expected.

Figure 1.5 shows a comparison of the *Set potential* command and the *CV staircase* command. The *Set potential* command has a single command parameter, *Potential (V)*, which defines the behavior of the whole command. The *CV staircase* command has a total of seven editable command parameters, two non-editable command parameters. Additionally, the *CV staircase* command also has a local sampler options. The editable command parameters must all be specified for the command to work.

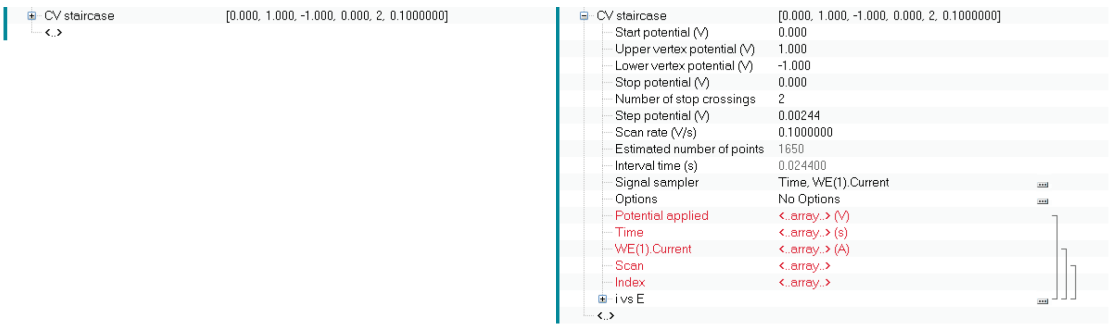


Figure 1.5 – Comparison of the *Set potential* command (left) and the *CV staircase* command (right)

> **Note**
>
> The command parameters for the *CV staircase* command are shown in different colors. More information on this color scheme is provided in Chapter 2 of this manual.

1.3.2 – The measurement sequence

A measurement in NOVA occurs according to a sequence of commands specified by the user in the **Procedure**. When the measurement starts, the procedure is always verified by the validation tool. If no errors are detected in the procedure validation tool, the measurement starts. During the measurement, the following basic steps are repeated:

- **Set Autolab property:** the instrument state is changed according to the parameters defined by the user in the procedure (Set potential, Set cell, apply next potential step, etc.).
- **Sample signals:** a change in the instrument state can trigger a reaction of the electrochemical cell. In order to monitor this reaction, the *electrochemical signals* are sampled, using the Signal sampler. The measured data can be displayed on a plot.
- **Options:** after the signals have been sampled, a user-defined set of options is used (Automatic current ranging, Cutoff condition, etc.).

These basic steps are repeated for each command in the procedure until the procedure ends or the user interrupts the measurement or a Cutoff condition is encountered. When the measurement stops, the measured data points can be stored into a user-defined database.

Figure 1.6 shows a schematic overview of the basic steps involved in a NOVA procedure.

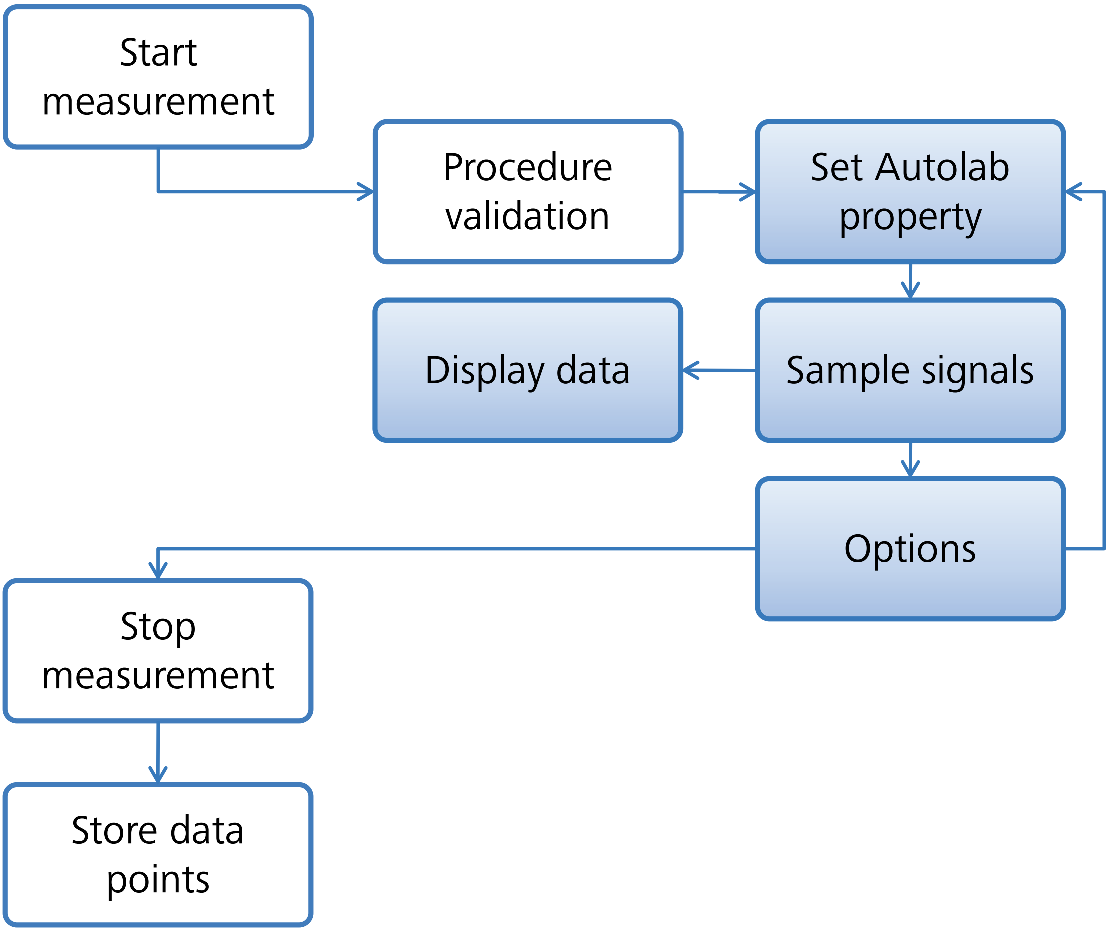


Figure 1.6 – The basic steps in a NOVA procedure

1.3.3 – The Signal sampler

The Signal sampler is a fundamental component of NOVA. The sampler defines what information (electrochemical signals) the Autolab instrument must gather during the course of an experiment. Typically, the Autolab will be used to measure both potential and current, but other signals can be acquired during a measurement, i.e. pH, time, temperature, QCM, etc. The sampler also defines how these signals must be measured (see Figure 1.7).

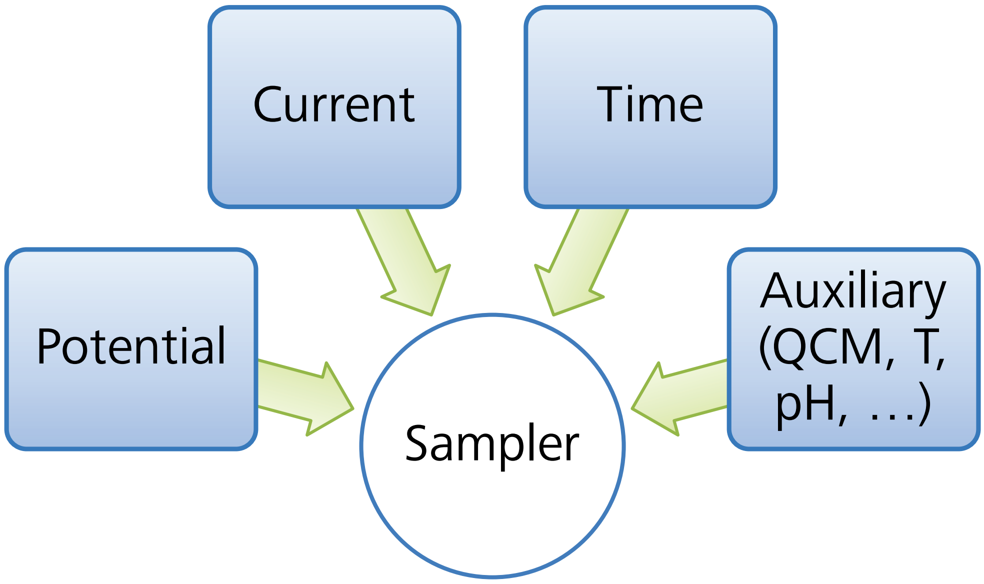


Figure 1.7 – The sampler is used to measure the electrochemical response of the cell

Every measurement command has a user-defined sampler. This means that individual measurement steps can be designed in order to gather data points from the relevant sources. The sampler is defined as a series of segments, during each of which one or more electrochemical signals are recorded. The user can decide if a specific electrochemical signal can be sampled only one time during a segment, or if an averaged value of several measurements is required.

The sampler can be edited by clicking the sampler button in the procedure editor (see Figure 1.8).

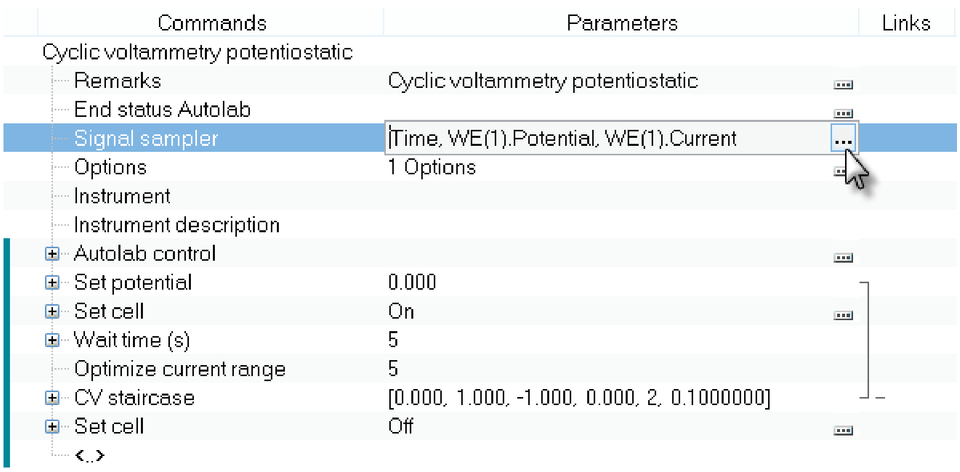


Figure 1.8 – Modifying the signal sampler for the cyclic voltammetry experiment

The signal sampler can also be opened by clicking the button in the quick access toolbar, when this button is shown (see Figure 1.9).

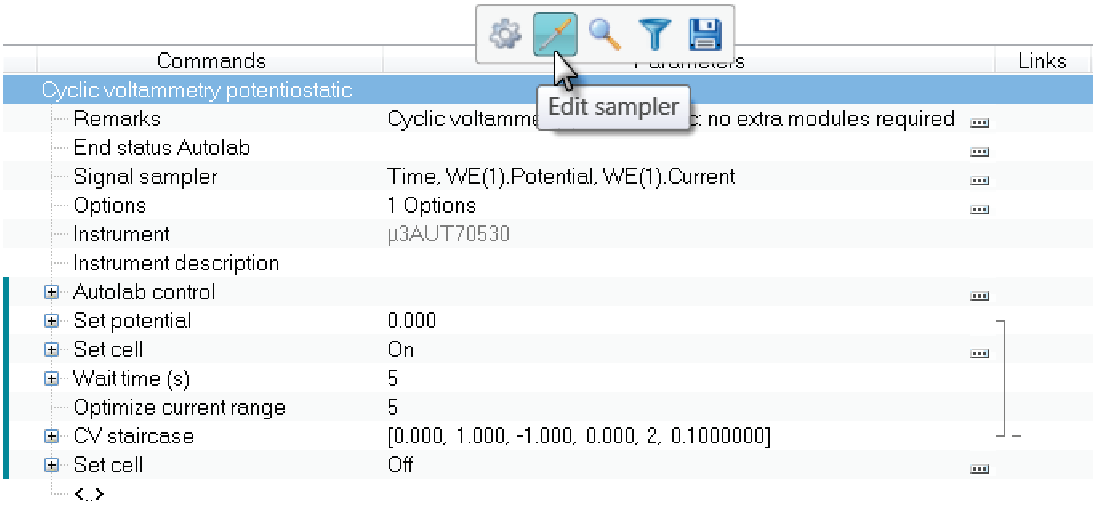


Figure 1.9 – Clicking the button in the quick access toolbar opens the Edit sampler window

The Edit Sampler window will be displayed. In this window, the available electrochemical signals displayed (see Figure 1.10). A checkbox is provided for each signal.

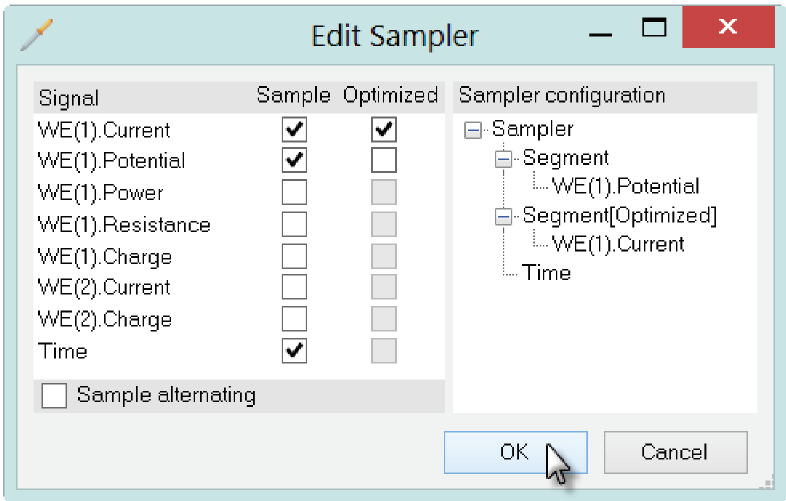


Figure 1.10 – The Sampler edit window

Figure 1.10 shows a sampler setup to measure time (Time), potential (WE(1).Potential) and current (WE(1).Current) during the measurement. Other signals are available, but are not sampled during the measurement (WE(1).Power, WE(1).Resistance, WE(2).Current and WE(2).Charge).

The WE(1).Current signal is also checked as *Optimized* in the sampler shown in Figure 1.10 whereas the Time and the WE(1).Potential signals are only checked once, in the Sample column.

An important difference exists between a sampled signal and a *sampled* and *optimized* signal.

When a signal is sampled, the instrument performs a single Analog-to-Digital (A/D) conversion for that signal. However, when a signal is also optimized, the measured value of a signal is an average value obtained from a large number of A/D conversions. Optimizing a signal improves the signal to noise ratio.

Note

It is only possible to optimize a signal if it is sampled. The Time signal cannot be optimized because of its discrete nature. WE(1).Power, WE(1).Resistance and WE(X).Charge are calculated values and cannot be optimized.

In the case of the sampler shown in Figure 1.10, the Time and WE(1).Potential are only sampled once at the beginning of the segment and then the WE(1).Current signal is sampled and optimized for a longer period of time, yielding an averaged value at the end of the segment (see Figure 1.11).

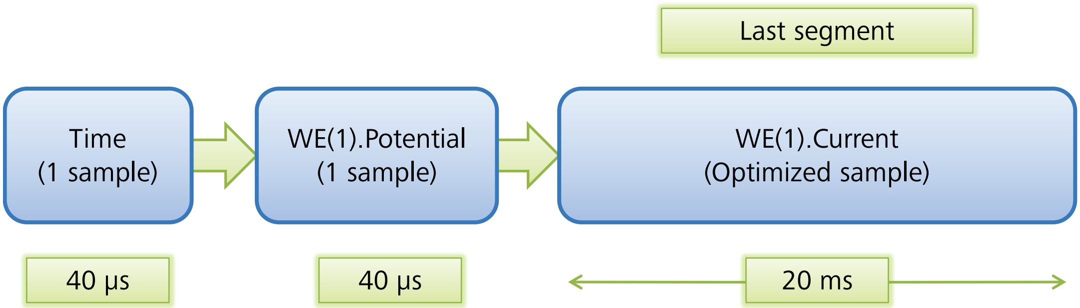


Figure 1.11 – A schematic view of the sampling process

The total time spent sampling Time and WE(1).Potential is roughly 40 µs[9] while the time spent sampling the WE(1).Current signal is about 20 ms or 16.67 ms[10], depending on the line frequency defined in the Hardware setup (50 or 60 Hz).

If an extra signal has to be sampled, for example the WE(2).Current provided by the BA module, it must be selected in the sampler window. This extra signal can then be sampled once just like Time and WE(1).Potential, or for a longer time, in which case it will be measured several times and averaged. Figure 1.12 shows the Edit Sampler window corresponding to this situation.

[9] This is the approximate duration of a single A/D conversion.
[10] Or the maximum available time when the interval time is smaller than 20 ms or 16.67 ms.

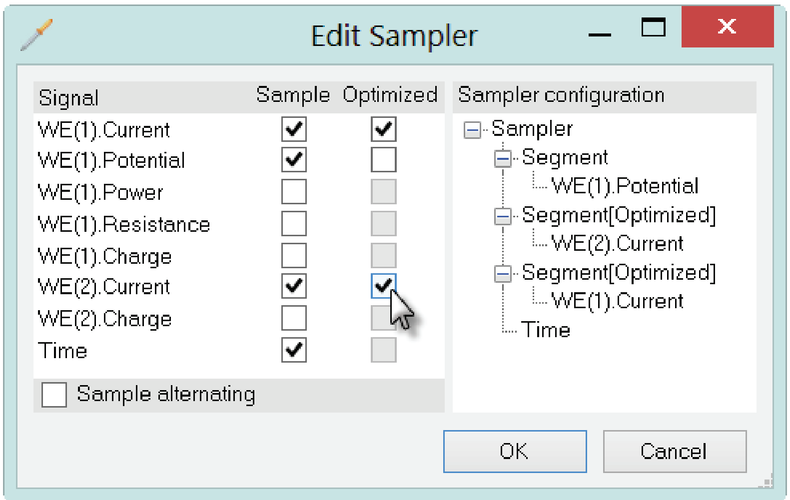


Figure 1.12 – Adding the WE(2).Current signal to the sample

If more than one signal is optimized, the total available time has to be shared. Figure 1.13 shows a schematic representation of this situation.

Note

The maximum number of signals can be measured *optimized* is **six**.

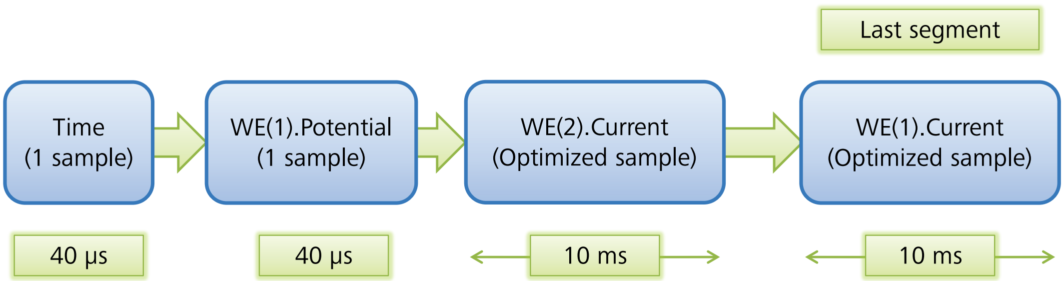


Figure 1.13 – Sampling an extra external signal

Note

The WE(1).Current signal is always sampled in the last sampling step.

Figure 1.14 shows a practical example of sampling an electrochemical signal, using a single A/D conversion for each data point or the optimized sampling method. The measured signal is the pH, provided by the pX module for the Autolab PGSTAT. Using the sample optimization reduces the noise pickup during the measurement.

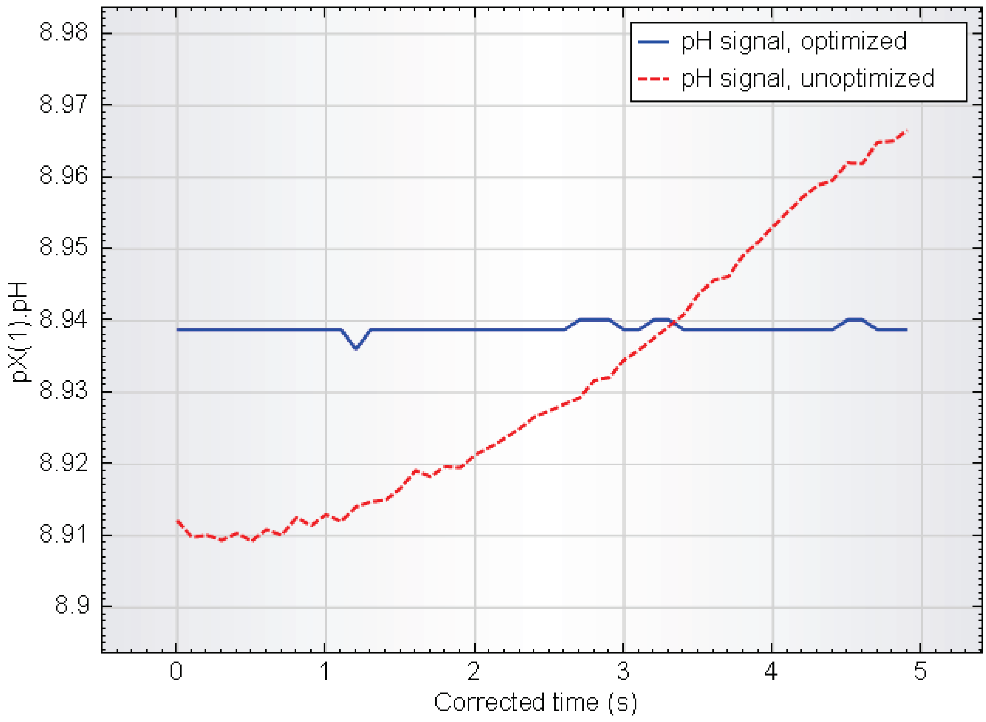


Figure 1.14 – A practical example showing the benefits of sampling a signal *Optimized*

If the WE(2).Current signal must be recorded in the same sampling segment as the WE(1).current, the *Sample alternating* option can be used (see Figure 1.15). Using this option, the WE(1).Current and the WE(2).Current signals will be sampled at the same time, in an alternating way.

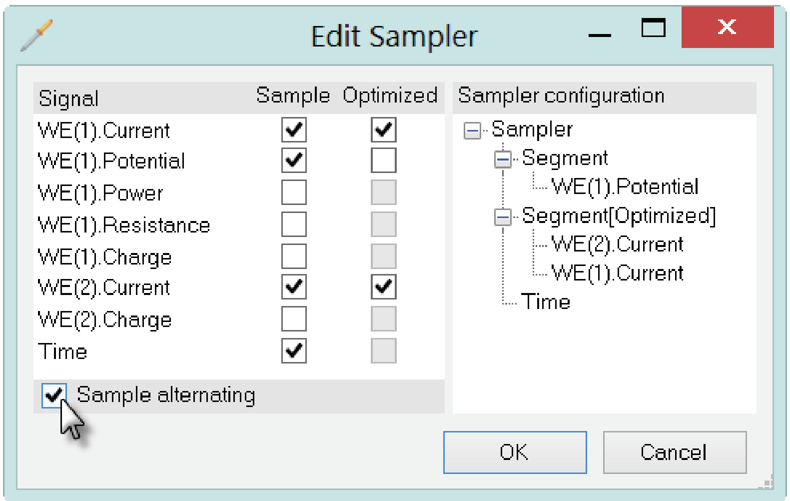


Figure 1.15 – The Sample alternating option can be used to sample signals in the same segment

The equivalent sampling time will still be the same as in the previous case, but using this option, both signals will be collected at the same time (see Figure 1.16).

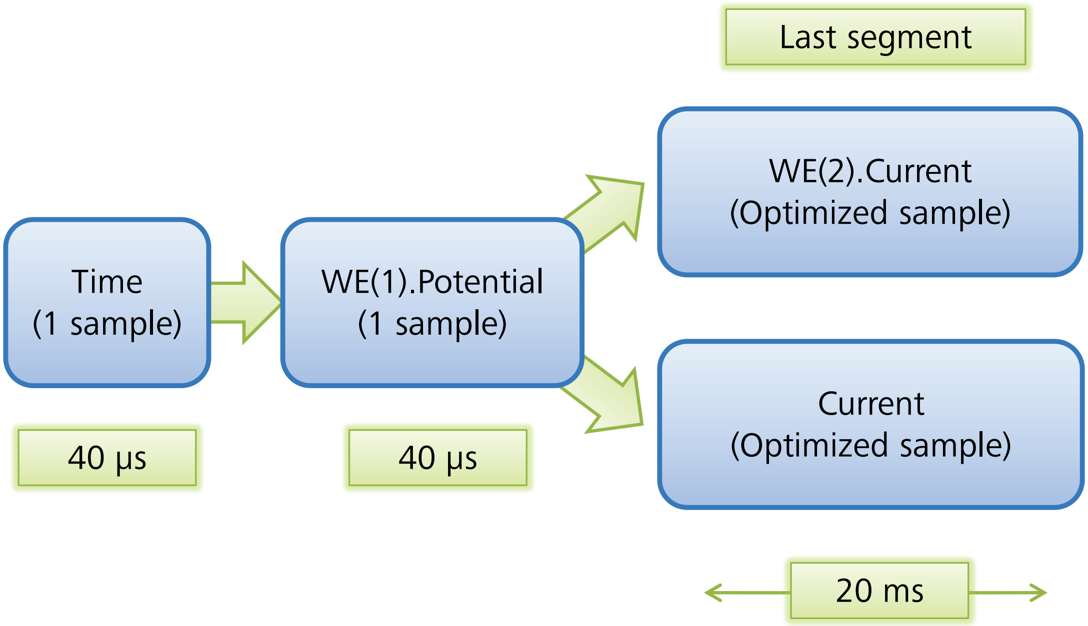


Figure 1.16 – Sampling an external signal using the sample alternating option

Note

Using the sampler lets the user define exactly which electrochemical signal to record during a measurement and when these signal have to be recorded.

1.3.4 – The Options

The options can be used at the very end of each sampling step to evaluate the measured data and change the data acquisition settings, test a cutoff condition or trigger a special action.

The following actions are part of the options:

- Automatic current ranging
- Cutoff[11]
- Autolab control[12]
- Automatic Integration Time[13]

The Options can be defined for each measurement command. To edit the options, click the options button ... in the procedure editor (see Figure 1.17).

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[11] For more information on the Cutoff option, please refer to the Cutoff tutorial, available from the Help menu.
[12] For more information on the Autolab control option, please refer to the Autolab control tutorial, available from the Help menu.
[13] For more information on the Automatic Integration Time option, please refer to the FI20 Filter and Integrator tutorial, available from the Help menu.

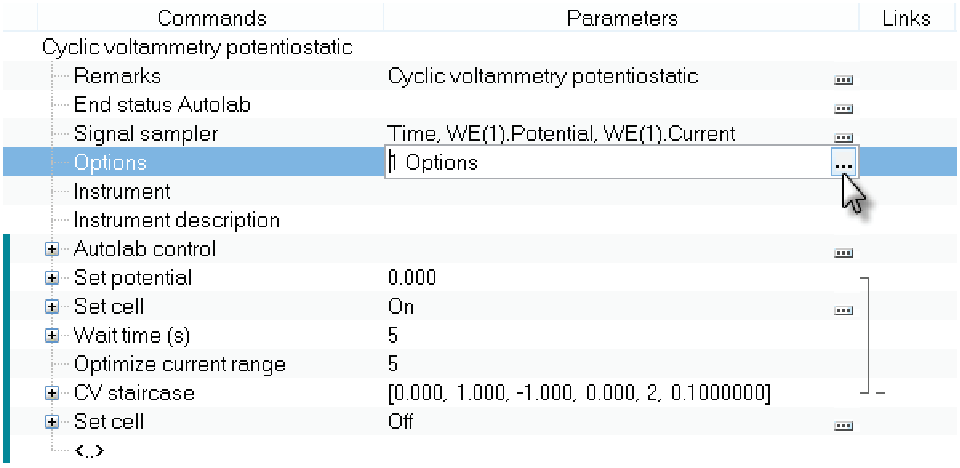


Figure 1.17 – Modifying the options for the cyclic voltammetry experiment

The options can also be opened by clicking the button in the quick access toolbar, when this button is shown (see Figure 1.18).

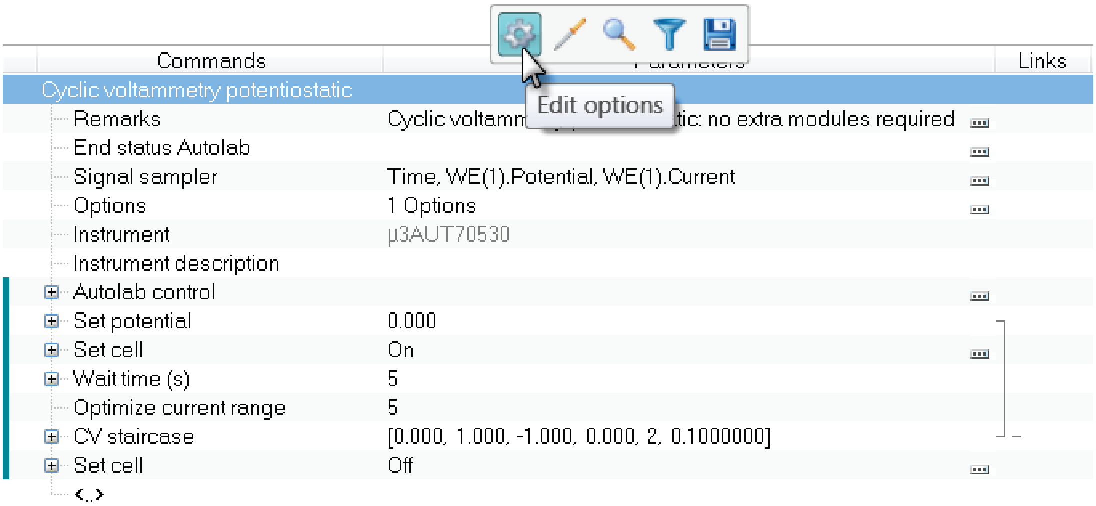


Figure 1.18 – Clicking the button in the quick access toolbar opens the Edit options window

The Edit Options window will be displayed (see Figure 1.19). In this window, the Automatic current ranging settings can be defined, for each working electrode (on the Automatic current ranging tab). Additionally, the Cutoff tab can be used to define cutoff conditions for the measured signals and the Autolab control tab can be used to define special actions for the Autolab instrument[14]. If an analog integrator is available, the Automatic integration time option can be specified in the final tab.

[14] For example, create a new drop on a Hg electrode in a polarographic measurements.

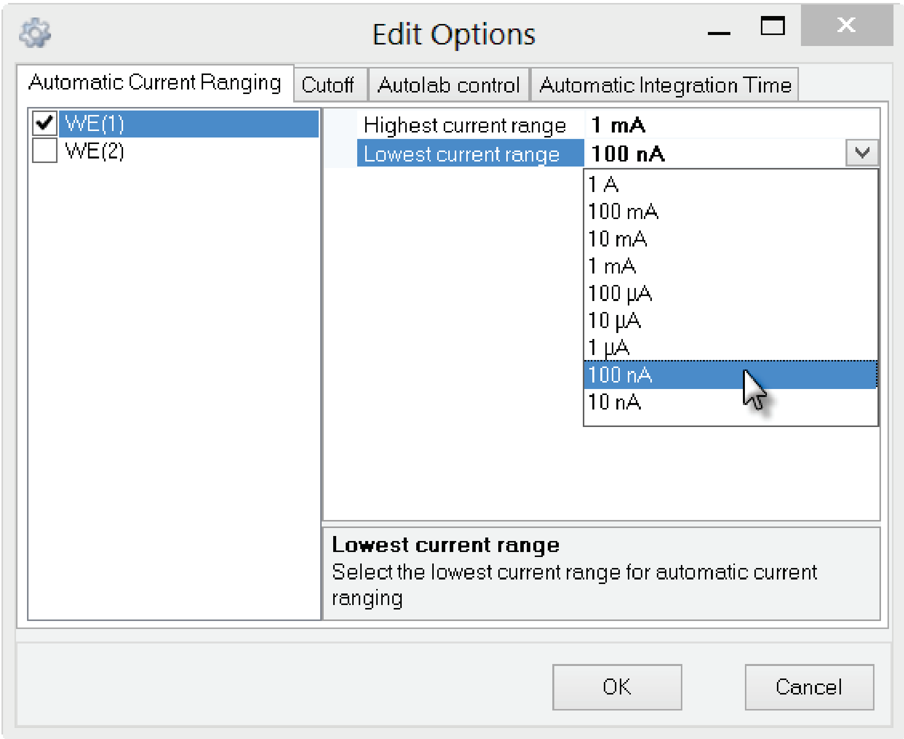


Figure 1.19 – The Edit Options window

If the Automatic current ranging option is used, the current range will be adjusted if necessary, at the end of the sampling step, based on the last recorded value of the current[15]. If the Cutoff option is used for a sampled signal, the last recorded value of this signal is tested against the threshold value defined in the cutoff. Specific actions can be triggered by the Cutoff[11] (Stop command, Stop complete procedure, etc.).

1.4 – Global and local sampler/options

The signal sampler and options detailed in the previous sections (see Sections 1.3.3 and 1.3.4) can be defined for the complete procedure and for each measurement command that supports the use of the signal sampler and/or options in the procedure.

When the sampler or options are defined for the whole procedure, these will be referred to as **global** properties. All the measurement commands included in the procedure will use the global sampler or global options.

When the sampler or options are defined for a single command in the procedure, these will be referred to as **local** properties. Only this single command in the procedure will use these properties and these will overrule the settings specified in the global sampler and options.

[15] The current range is set one range higher when the measured current is > than 3 times the active current range. The current range is set one range lower when the measured current is < than 0.3 times the active current range.

Figure 1.20 shows an example of global and local options for the cyclic voltammetry potentiostatic procedure. The *CV staircase* command has been edited and the WE(2).Current signal has been added to its local sampler.

Commands	Parameters	Links
Cyclic voltammetry potentiostatic		
Remarks	Cyclic voltammetry potentiostatic	
End status Autolab		
Signal sampler	Time, WE(1).Potential, WE(1).Current	
Options	1 Options	
Instrument		
Instrument description		
Autolab control		
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]	
Start potential (V)	0.000	
Upper vertex potential (V)	1.000	
Lower vertex potential (V)	-1.000	
Stop potential (V)	0.000	
Number of stop crossings	2	
Step potential (V)	0.00244	
Scan rate (V/s)	0.1000000	
Estimated number of points	1650	
Interval time (s)	0.024400	
Signal sampler	Time, WE(1).Potential, WE(2).Current, WE(1).Current	
Options	1 Options	
Potential applied	<..array..> (V)	
Time	<..array..> (s)	
WE(1).Current	<..array..> (A)	
Scan	<..array..>	
WE(1).Potential	<..array..> (V)	
WE(2).Current	<..array..> (A)	
Index	<..array..>	
i vs E		
Set cell	Off	
<..>		

Figure 1.20 – The Cyclic voltammetry potentiostatic procedure (local sampler defined for the *CV staircase* command)

1.5 – Data management

Data storage in NOVA is done by means of a user-defined database. In practice, this means that all the measurements are stored in a single folder, without user intervention (either when the experiment is finished or if the measurement is interrupted). Using a database for data storage offers four major advantages compared to a file based storage system:

1. Everything is stored in a single, structured, folder.
2. Making backups of experimental data becomes very easy.
3. Filters allow fast recovery of experimental data.
4. The database provides a repository for easy data restoration.

A database manager is provided in NOVA and it can be accessed by clicking on the **Tools** menu and clicking the Database Manager option (see Figure 1.21).

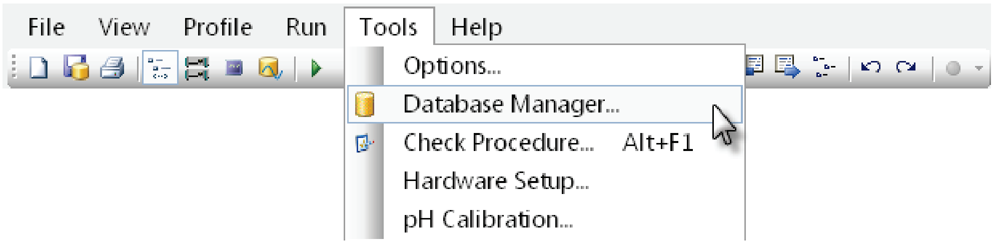


Figure 1.21 – Accessing the Database Manager

The database management window will appear, displaying the path for four different pre-defined databases (see Figure 1.22). Each database is located in a folder on the computer.

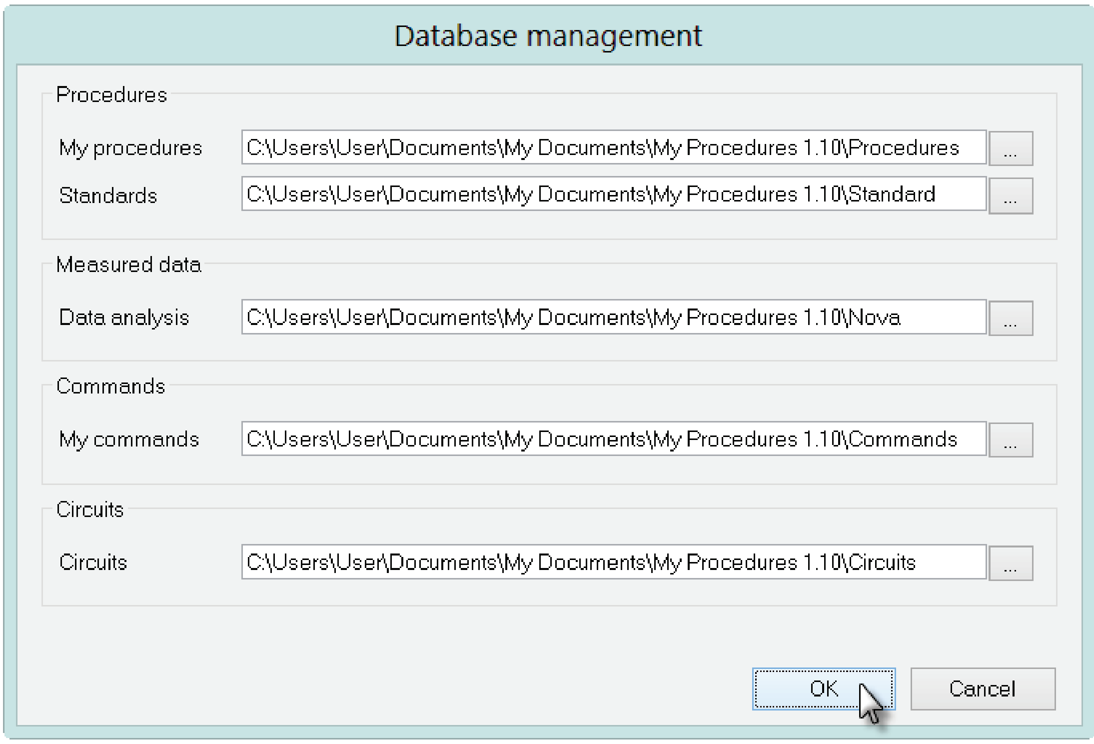


Figure 1.22 – The database management window with the four default databases

The database management window displays the locations of two *Procedures* databases (Standard and User), a *Measured data* database (Data), a *Commands*[16] and a *Circuits*[17] database.

The User database is the database where all the user-defined procedures are stored while the Data database is where all the measured data sets are recorded.

Note

The **Standard** database is intended to be used as a second *read-only* database. If the path of this database points to a database containing some user-defined procedures, those procedures will appear under the standard group in the procedure browser frame but will have a read-only status like the procedure displayed under the Autolab group in the procedure browser frame. In practice, any modification of a procedure from the Standard group can only be saved in the My Procedures group.

[16] The Commands database is used to store user defined NOVA commands. Please refer to Chapter 2 of this User manual for more information.

[17] The Circuit database is used to store equivalent circuits used in the fitting and simulation of electrochemical impedance spectroscopy data. Please refer to the impedance spectroscopy tutorial for more information.

Note

Nova gives the user the possibility to create any number of databases. A convenient way to manage your experiments is to create a new database every week, or create a specific database for each type of experiment. The database can also be located on a mobile storage device like a USB stick, an external hard drive or on a network drive.

To change a database, click the button on the right of area displaying the path of the currently used database. This will open a browser interface, which can be used to select another database folder.

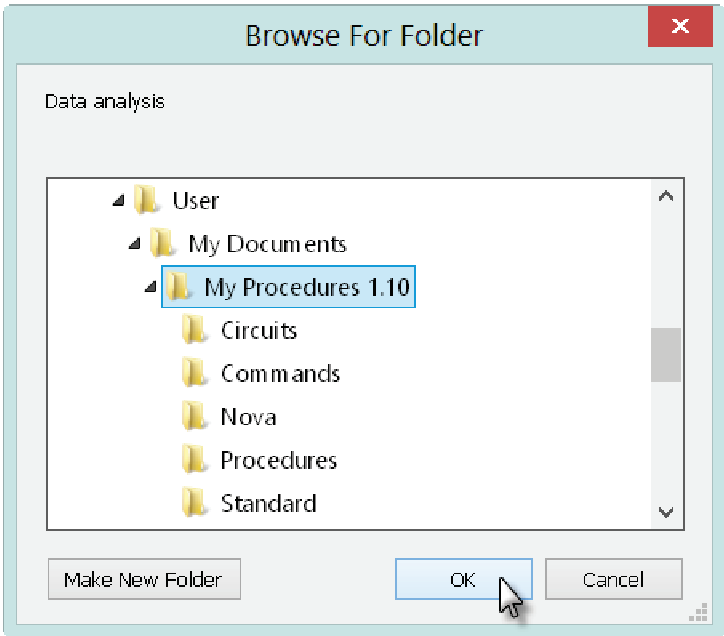


Figure 1.23 – A file browser window is used to change the location of the database folder

The database browser window can be used to create a new database by clicking the Make New Folder button. This will create a new folder, which can be renamed and used as a new database (see Figure 1.24).

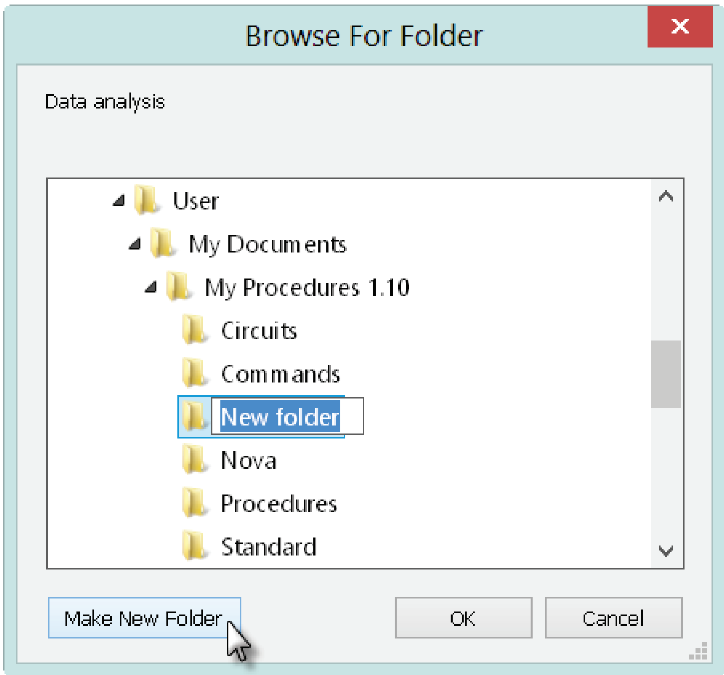


Figure 1.24 – Creating a new database

Note

It is possible to rename existing database folder by right-clicking the folder and selecting the Rename option from the context menu. The same menu can be used to delete a folder.

1.6 – Customizing NOVA

Two different customization options are provided in NOVA:

- **Profiles:** this option is used to filter the contents displayed in NOVA according to one or more profiles. The software comes with a number of factory default Profiles, but it is possible to create additional user-defined profiles. More information is provided in Section 1.6.1.
- **Software options:** this is used to define more advanced software options, like the default plot options, the Autolab display and the saving mode of the data files. More information is provided in Section 1.6.4.

1.6.1 – Profiles

A profiling scheme is available in NOVA. Using this tool, it is possible to hide some commands in order to simplify the user interface. Depending on the active profile, some commands or procedures are hidden from view in order to filter out the most relevant information. The profiles can be adjusted using the dedicated Profile menu (see Figure 1.25).

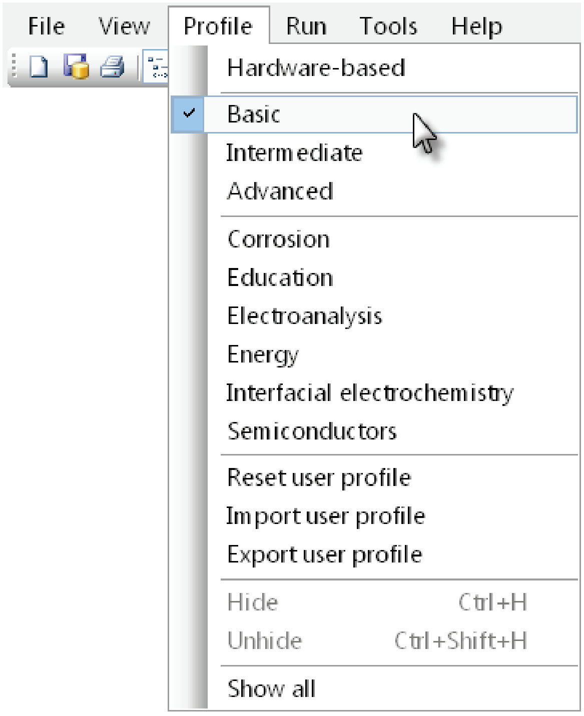


Figure 1.25 – The profile menu can be used to set the user profile

The profiles are grouped in three sections:

- **Level based (mandatory, set to Intermediate by default)**
- **Hardware based (optional)**
- **Application based (optional)**

The active profile or combination of profiles is indicated in the status bar of NOVA, in the lower right corner of the screen. Figure 1.26 shows that the Intermediate profile and the Hardware-based profiles are currently active.

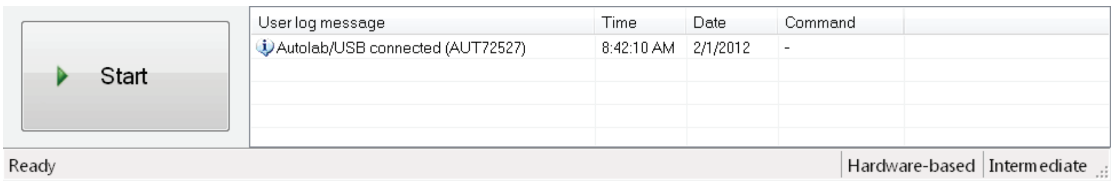


Figure 1.26 – The active profile or combination of profiles is indicated in the status bar of NOVA

1.6.1.1 – Level based

All the commands and the procedures in NOVA have been tagged with three levels: basic, intermediate and advanced. When the basic profile is active, only a few simple commands are shown, when the advance profile is active, all the commands are shown (see Figure 1.27).

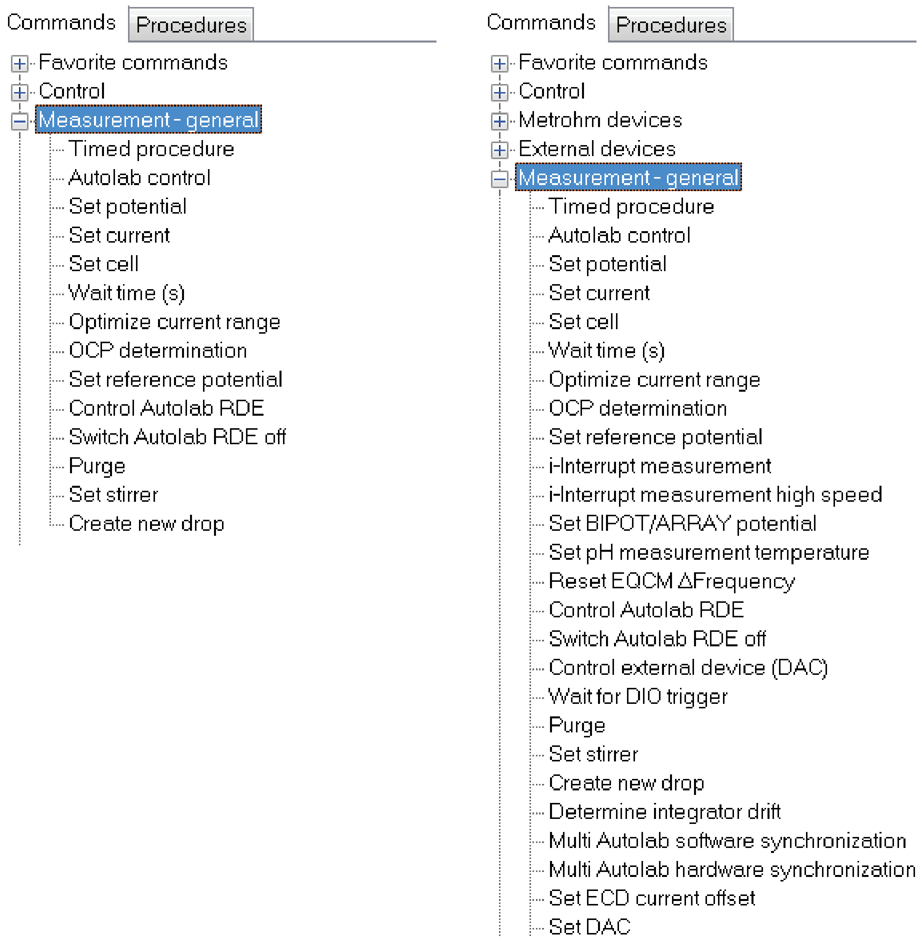


Figure 1.27 – Difference between the basic (left) and the advanced (right) profile

Note

The intermediate profile is the default level based profile selected upon installation of Nova.

Important

The selected level based profile is always on.

1.6.1.2 – Hardware based

All the commands and the procedures in NOVA have been tagged with a hardware requirement marker. When the hardware-based profile is active, only the procedures and commands that can be executed with the connected instrument are shown. In Figure 1.28, the Autolab procedures are shown without and with the hardware-based profile active, for a µAutolabIII. Since the µAutolabIII is not fitted with the required hardware, several procedures are hidden.

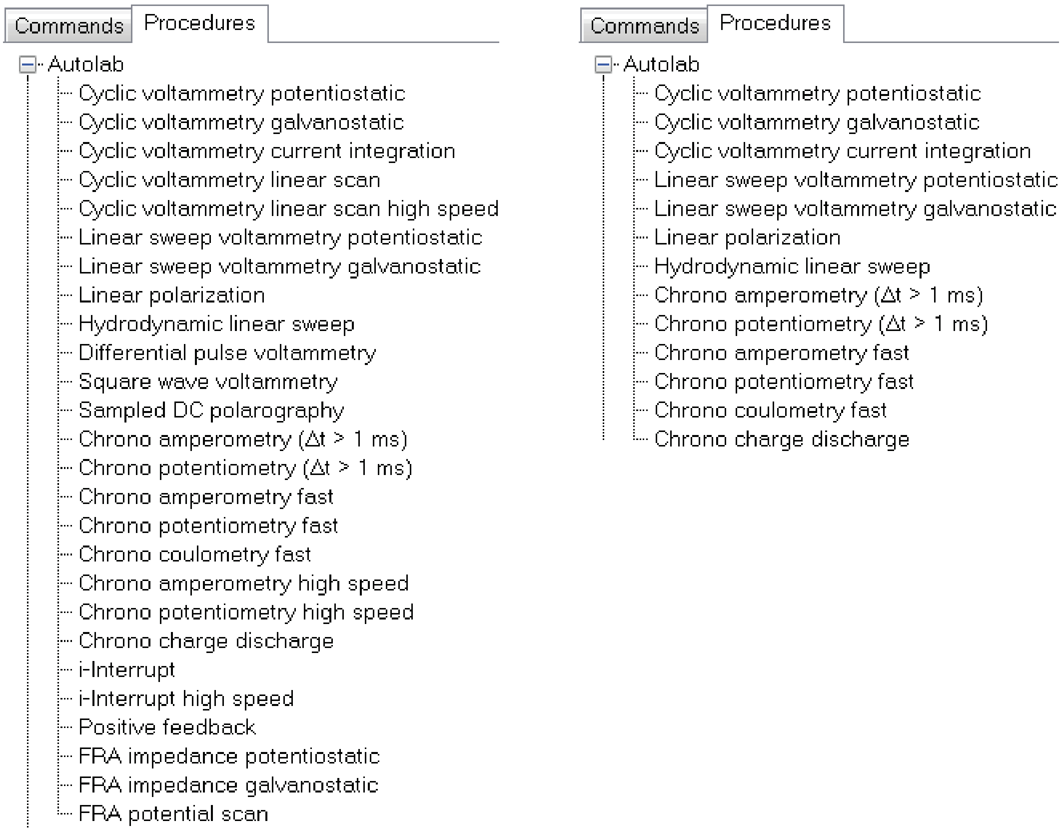


Figure 1.28 – Overview of the Autolab procedures with a µAutolabIII (left, hardware-based profile not active and right, hardware-based profile active)

1.6.1.3 – Application based

All the commands and the procedures in NOVA have been tagged with an application marker: corrosion, energy, electroanalysis, etc... When a given application profile is active, only the commands and procedures relevant for this application are shown (see Figure 1.29).

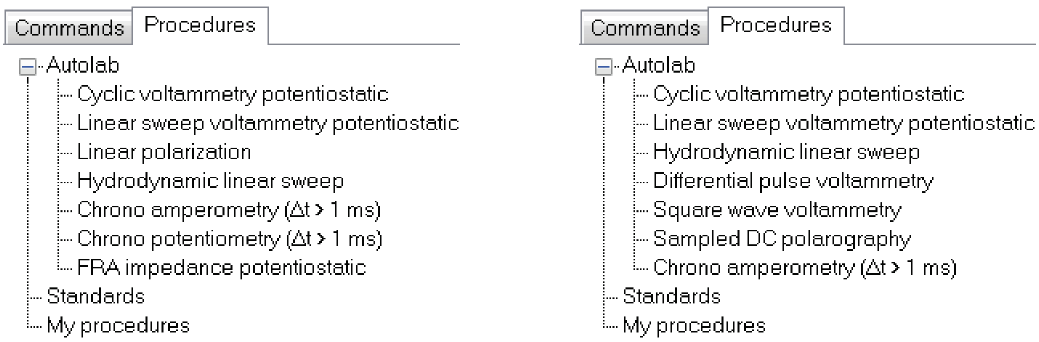


Figure 1.29 – Difference between the corrosion (left) and the electroanalytical (right) profile

Note

It is possible to combine all the profile schemes at the same time. For example, it is possible to set the profile to show hardware-based and intermediate commands and procedures for the energy application (see Figure 1.30).

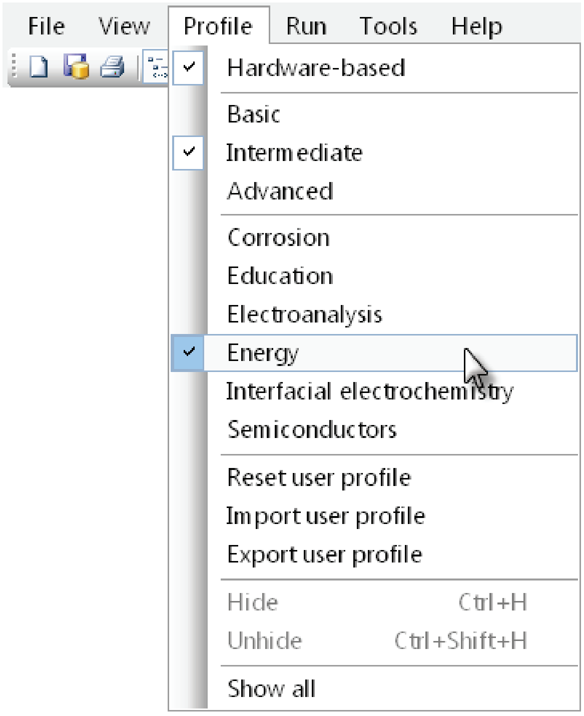


Figure 1.30 – It is possible to combine several profiles at the same time (intermediate, hardware-based and energy related)

1.6.1.4 – Hide/Show option

To further customize the appearance of the user interface, the option to hide and show procedures, commands, and command parameters has been added. To hide an item in NOVA, simply right-click the item and select the hide option from the menu. In Figure 1.31, this option is used to hide the *Chrono coulometry fast* procedure from the procedures browser.

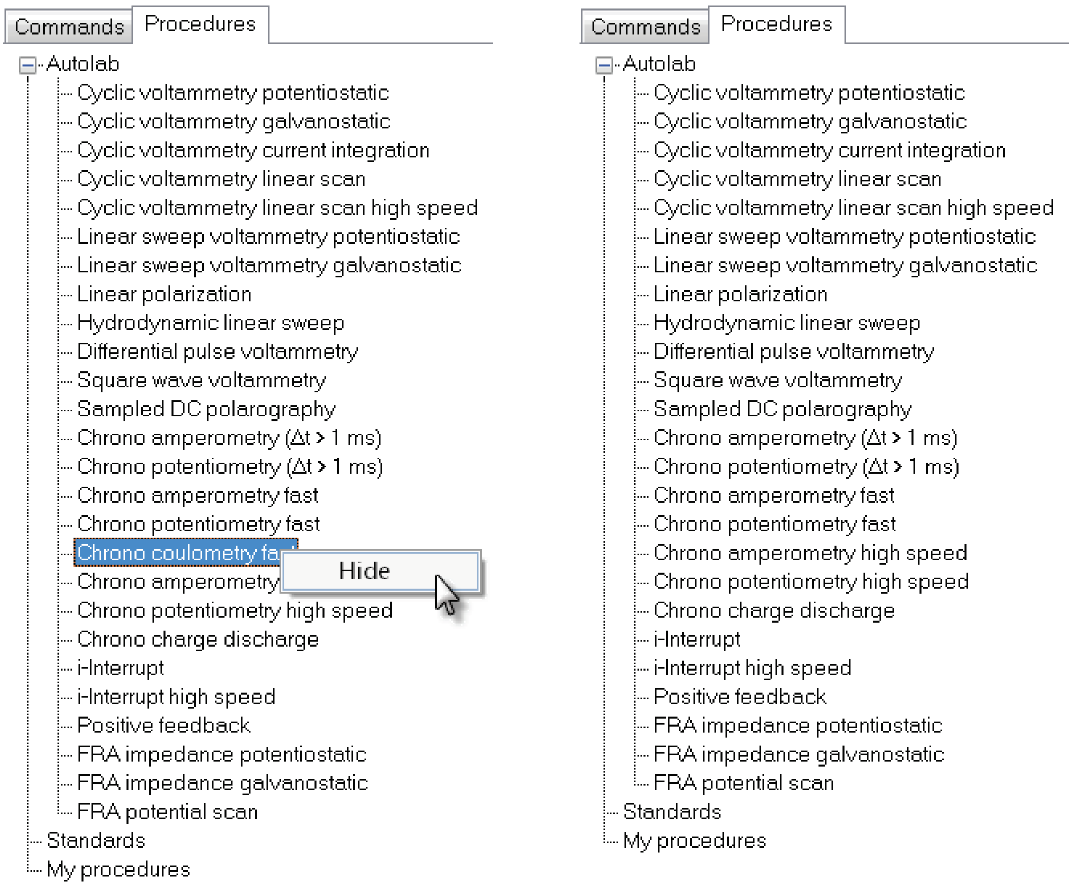


Figure 1.31 – Hiding a procedure from the procedure browser

Once an item has been hidden from view, it will not be shown until it is unhidden. To unhide hidden commands, procedures or command parameters, select the Show all option from the Profiles menu (see Figure 1.32).

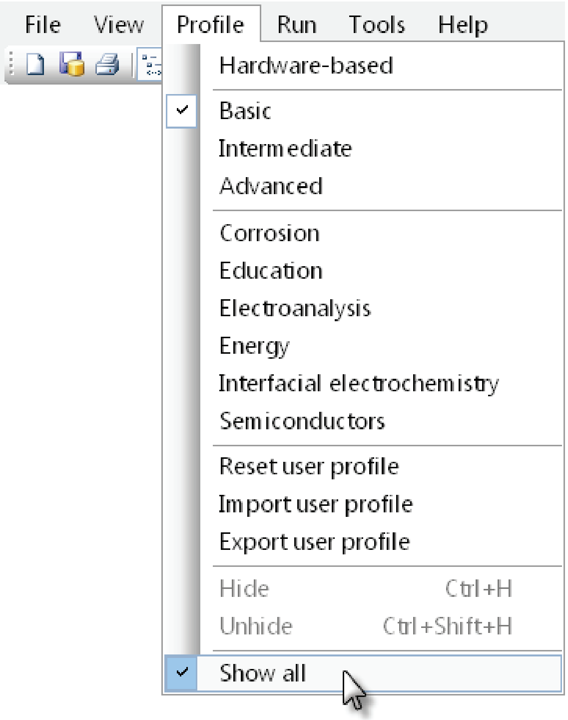


Figure 1.32 – Select the Show all option from the Profile menu to display all hidden items

Hidden items are shown in green (see Figure 1.33).

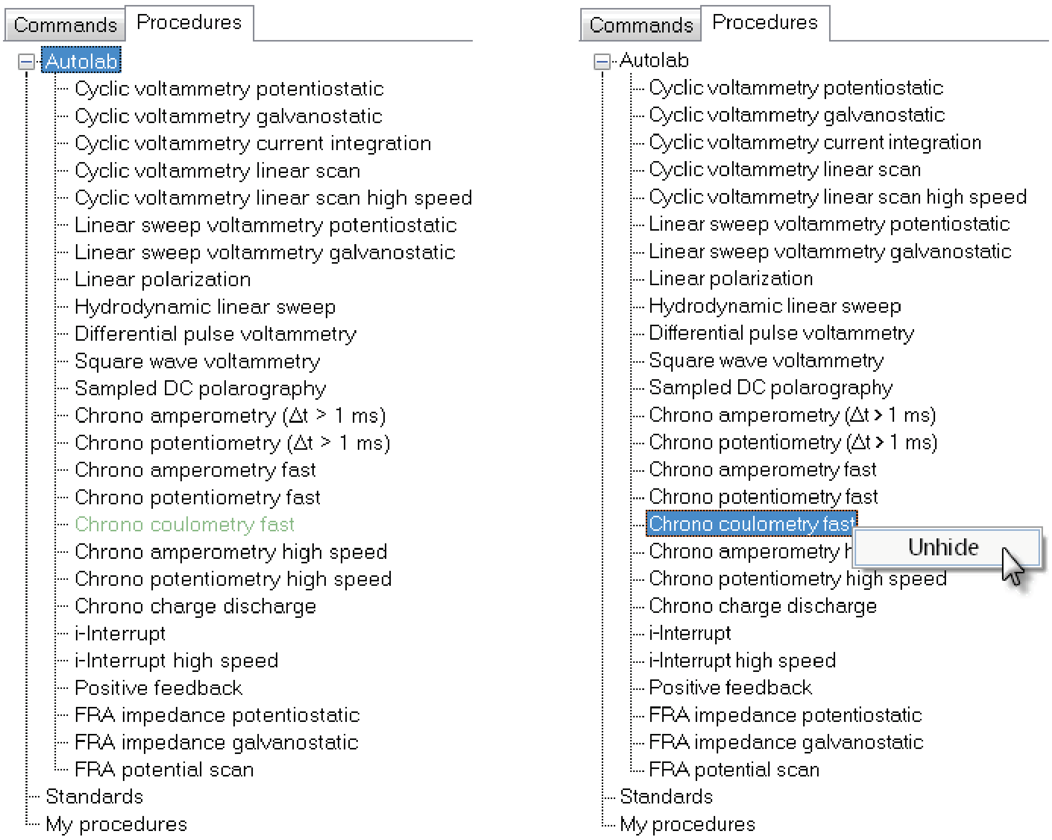


Figure 1.33 – Right click hidden items to unhide them

To unhide a hidden item, right-click the item and select the unhide option from the context menu, as shown in Figure 1.33.

Note

It is also possible to hide items using the CTRL-H keyboard shortcut and to unhide items using the CTRL-SHIFT-H keyboard shortcut.

In the commands browser, it is possible to hide single commands from a given group, using the same method. It is however also possible to completely hide a command group from the browser (see Figure 1.34).

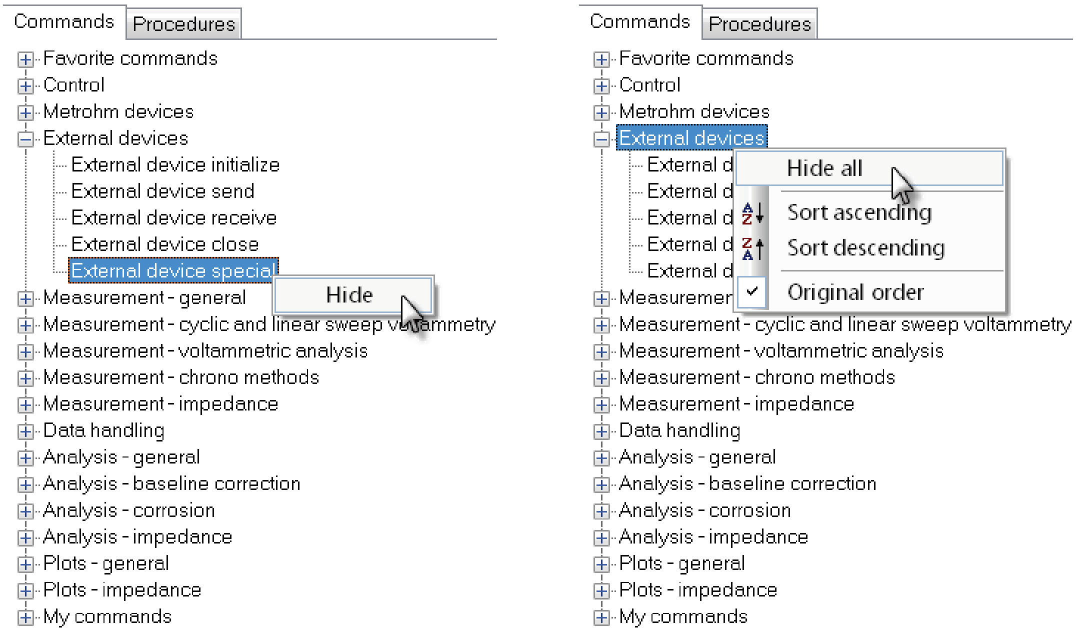


Figure 1.34 – Hiding a command (left) and a command group (right)

When a command group is hidden, the whole group will become invisible.

Finally, in the procedure editor, it is possible to hide or unhide command parameters, using the same method (see Figure 1.35). For example, if the interval time is not important, it can be hidden from view. This can be used to further simplify the user interface.

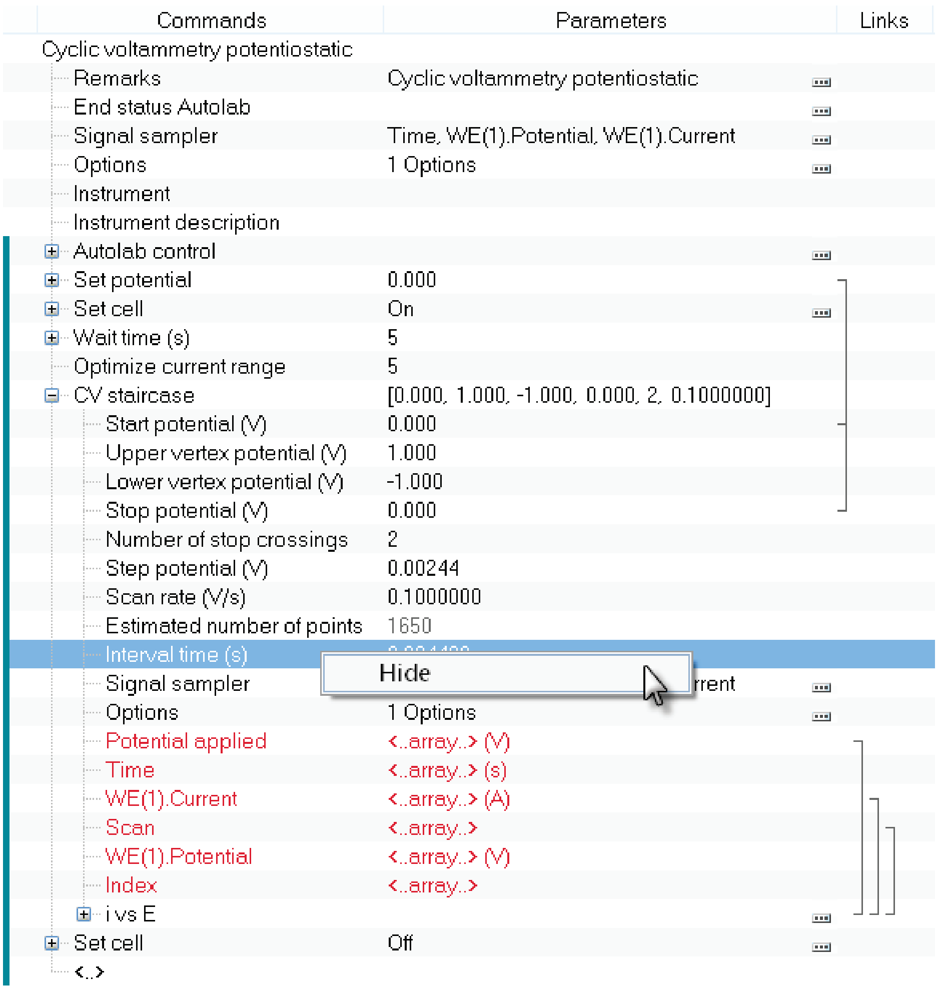


Figure 1.35 – Hiding and unhiding command parameters in the procedure editor

1.6.1.5 – Hidden commands warning

A warning is displayed in the status bar to inform the user when commands are hidden by the active profile in the procedure editor. This message will blink a few times (see Figure 1.36).

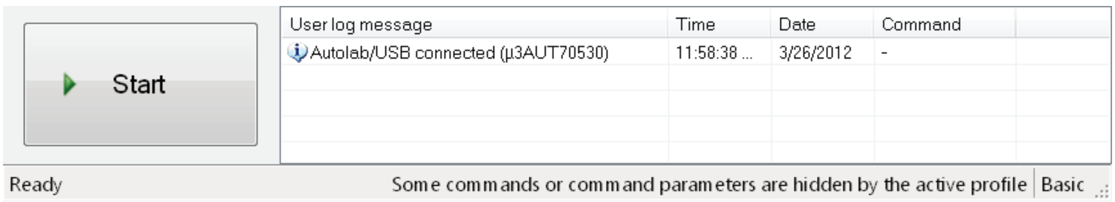


Figure 1.36 – A warning is displayed when commands are hidden by the active profile

To reveal the hidden commands, select the Show all option from the Profile menu or adjust the active profile using the same menu.

1.6.2 – Sorting function for the commands browser

The possibility to sort the commands in a group is now provided. Right-clicking a command group displays the different sorting options: ascending or descending (see Figure 1.37). It is always possible to return to the original order using the same strategy and using the Original order option provided in the context menu.

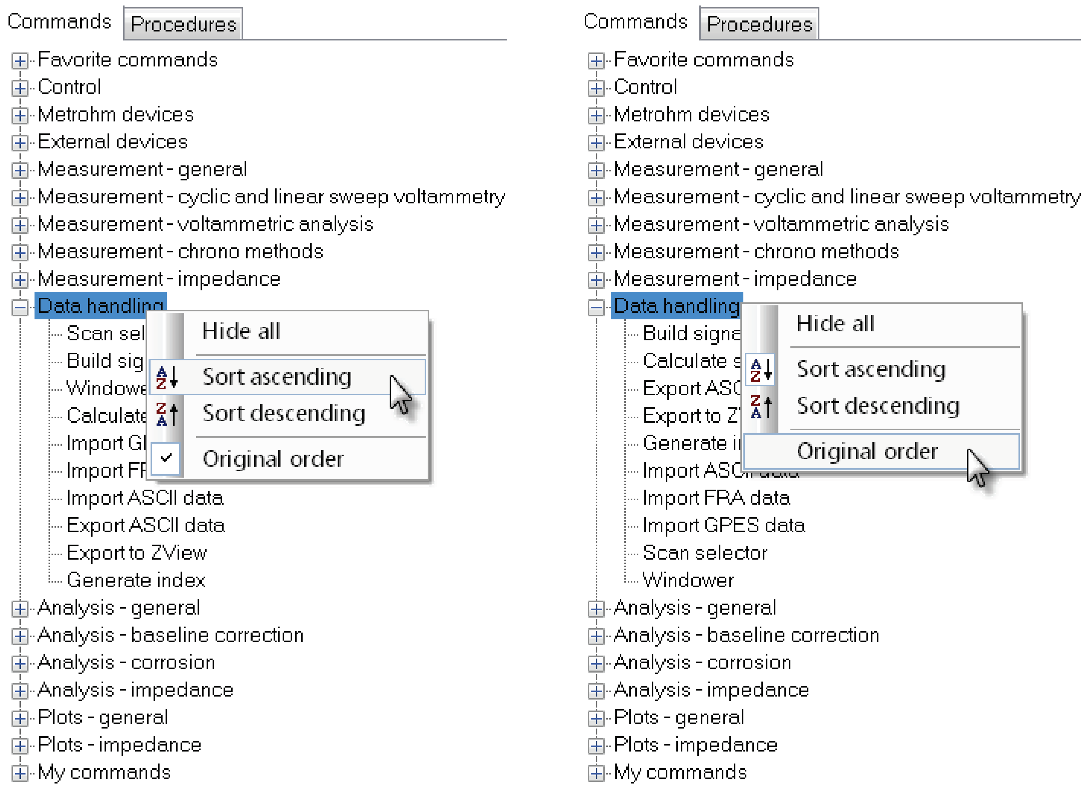


Figure 1.37 – Sorting the commands in a group in the commands browser

1.6.3 – Import/Export profiles

It is possible to hide any number of items in NOVA and to create a new profile for this customized view. Using the Profile menu, it is possible to import and export user profiles (see Figure 1.38).

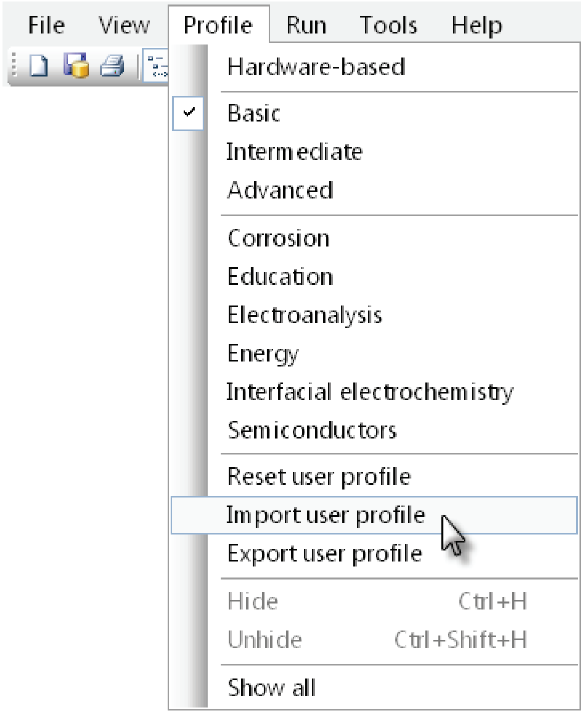


Figure 1.38 – Importing and exporting user profiles

This means that any number of profiles can be created and used at any time using this method.

It is possible to reset the user-defined profiles to default by using the Reset user profile option from the Profile menu (see Figure 1.38). A confirm message will be displayed when the user profile is reset (see Figure 1.39).

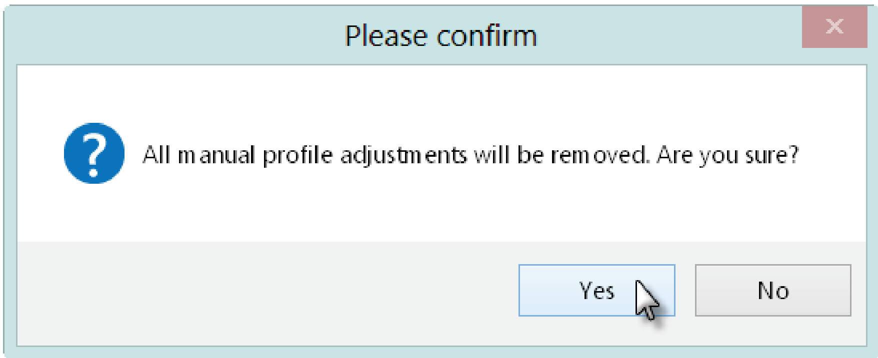


Figure 1.39 – A confirmation message is displayed with the user settings are reset

1.6.4 – Software options

It is possible to customize the behavior of NOVA, through the Options window, which can be accessed through Tools menu (see Figure 1.40).

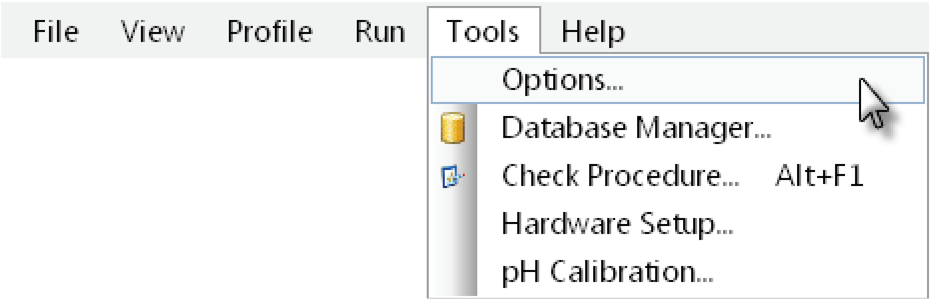


Figure 1.40 – Customization of NOVA can be done through the Options

Selecting the Options displays the NOVA options window (see Figure 1.41). Three different groups are shown in the left-hand side panel of the window:

- **Graphics**
- **Autolab display**
- **Advanced settings**

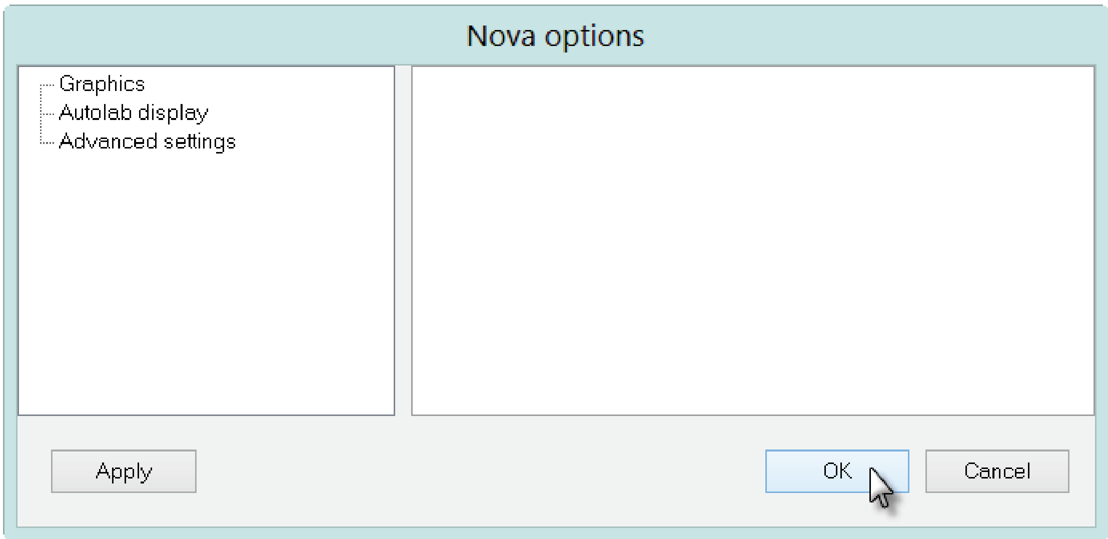


Figure 1.41 – The NOVA options window

1.6.4.1 – Graphics

In this category, the settings are grouped in two sections (see Figure 1.42):

- **General:** settings related to the plotting of the data.
- **Measurement view:** settings related to the number of points displayed in the measurement view during a measurement.

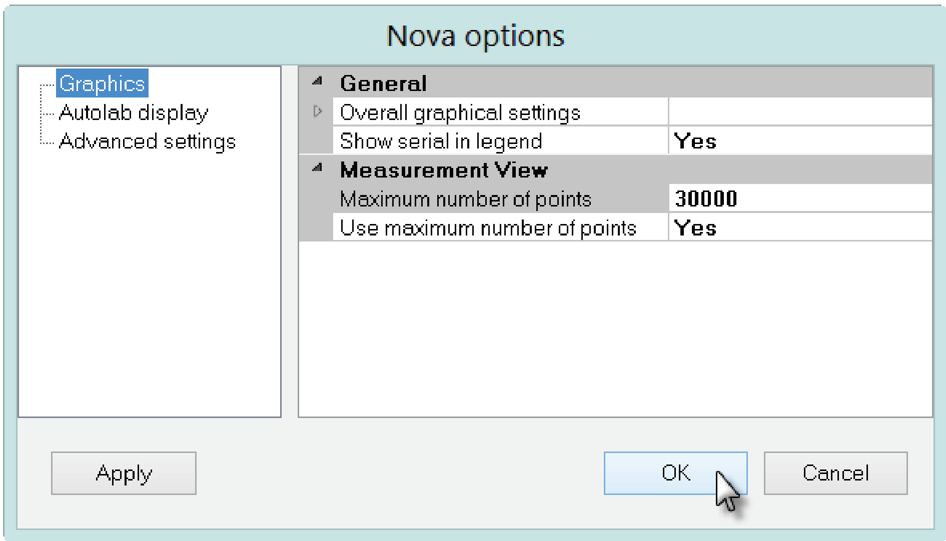


Figure 1.42 - The options listed in the Graphics section

1.6.4.1.1 – Default graphical settings

The default graphical settings can be defined by clicking the ... button next to the overall graphical settings to open the plot options window (see Figure 1.43).

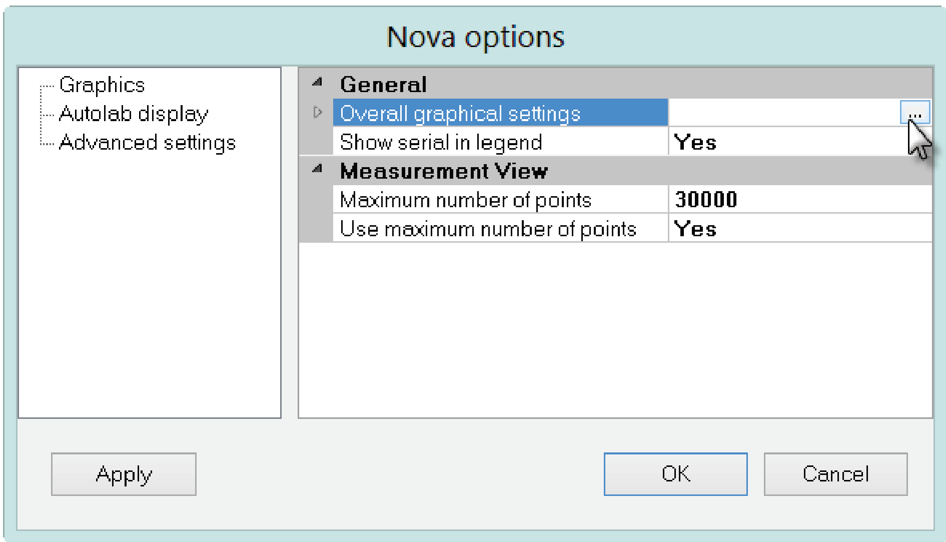


Figure 1.43 – The default plot options can be defined in the NOVA options

The Plot Options window will be displayed (see Figure 1.44). This window can be used to define the default settings used for all the plots in the software.

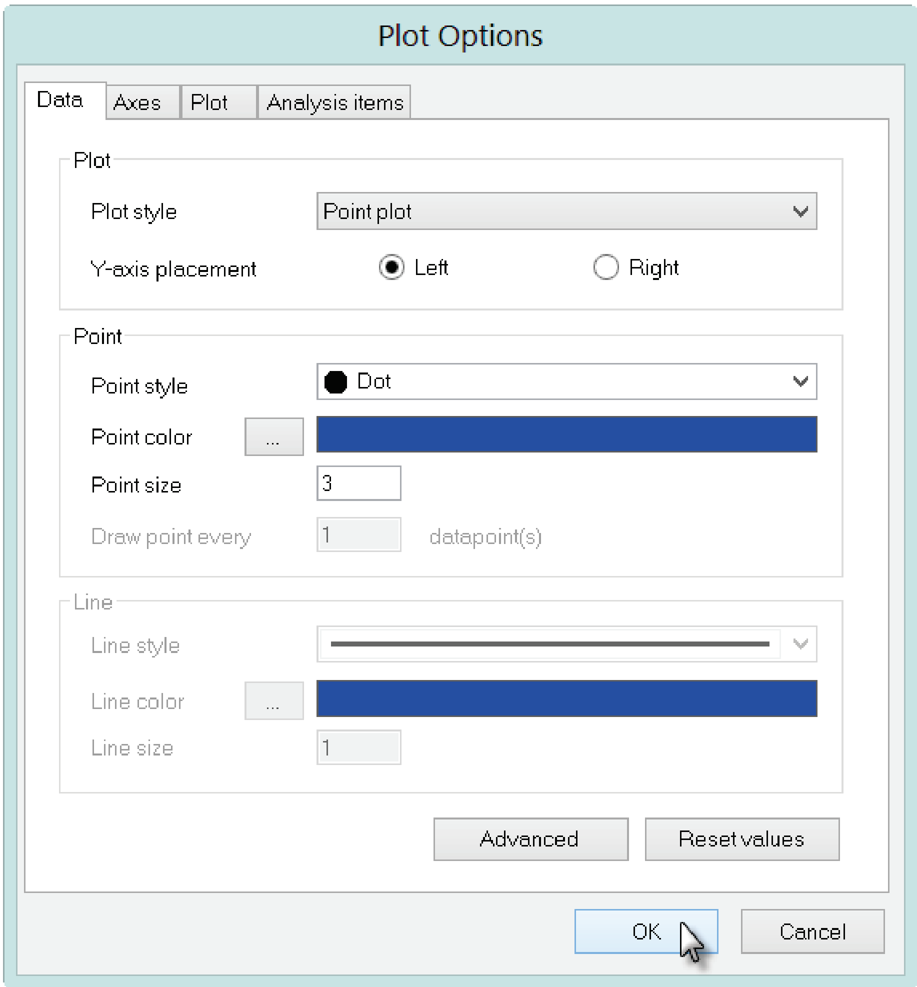


Figure 1.44 - The default plot options can be defined

Four tabs are available:

- **Data:** these settings define the overall aspect of the plots (point plot, line plot, etc.)
- **Axes:** these settings define the type of axis to use in the plots (linear, logarithmic, etc.)
- **Plot:** these settings are used to define the title, the background style, grid format, etc.
- **Analysis items:** these settings define the way data analysis items should be displayed on the plots

More information on plot settings can be found in Chapter 4 of this user manual (see section 4.2.2).

1.6.4.1.2 – Plot Legend settings

By default, the Autolab serial number is displayed in the legend of any plot to facilitate the identification of the instrument in an overlay plot. It is possible to deactivate this option through the NOVA options (see Figure 1.45).

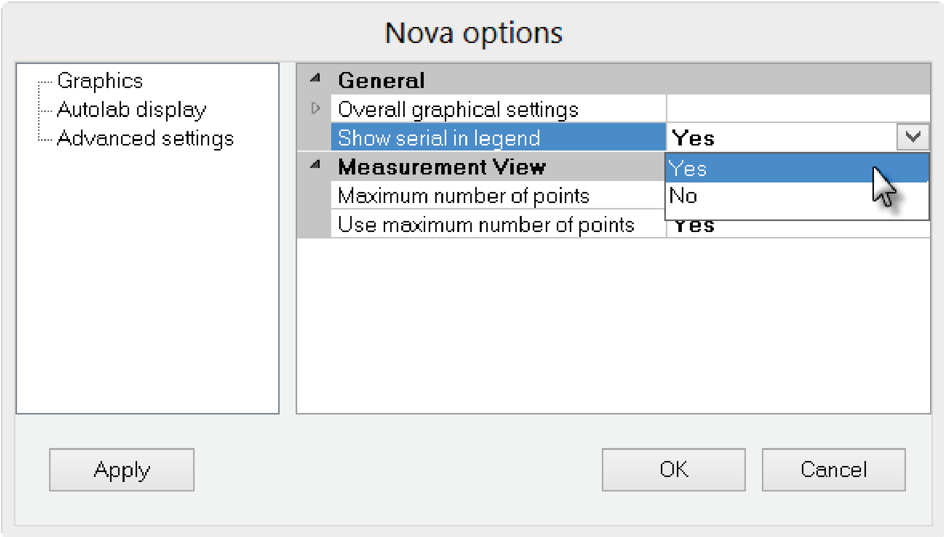


Figure 1.45 – The NOVA options allow the serial number to be activated or deactivated in the legend

Figure 1.46 shows an example of plot legends with and without the serial number of the instrument displayed.

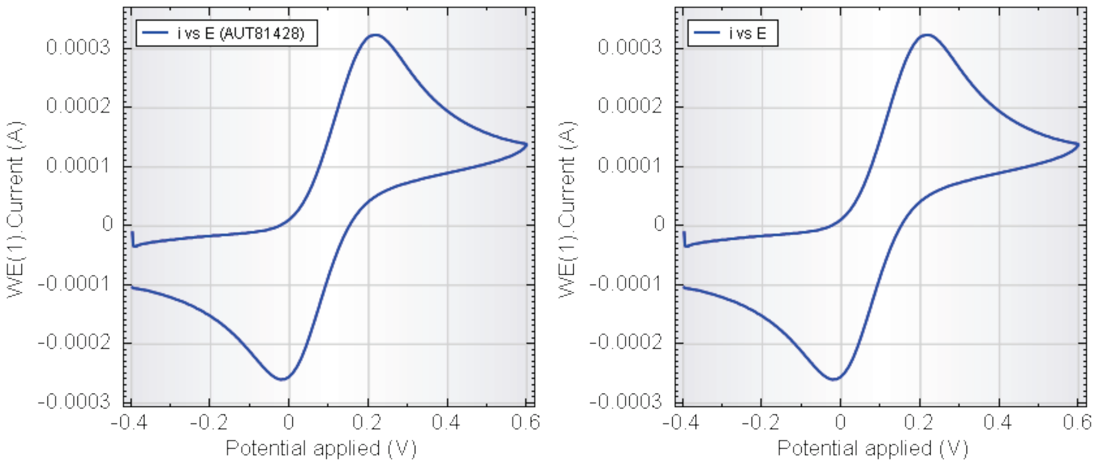


Figure 1.46 – Plot legend with (left) and without (right) the serial number of the instrument displayed

1.6.4.1.3 – Maximum number of points in real time view

Individual plots shown in the measurement view are limited to a fixed value 10000 points. A circular plotting buffer is used to plot data points, which means that when more than 10000 points are plotted in real time, a running buffer of 10000 points will be shown.

When a measurement command is finished, the last 10000 points collected by this command will remain displayed in the plot area and the next measurement command will start adding points to the plot, if applicable.

The *Maximum number of points* setting can be used to define the maximum number of points to show in the real time view during a measurement, for each of the four plots areas. The default value is 30000 (see Figure 1.47).

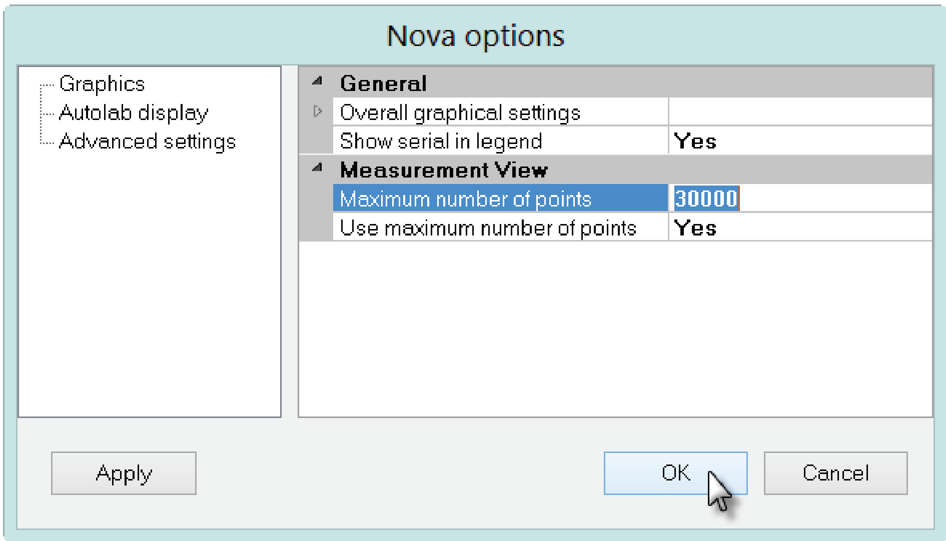


Figure 1.47 – The maximum number of points in real time view

If the **total** number of points shown in one of the four plot areas in the measurement view exceeds the value defined for the Maximum number of points setting, data points from finished commands will be removed in chronological order.

Important

A smaller number of points will increase the update rate of the plots during a measurement and reduce the memory usage of the computer. A larger number of points will decrease the refresh rate and increase memory usage.

This setting is overruled by the *Use maximum number of points in real time view* (see Figure 1.48).

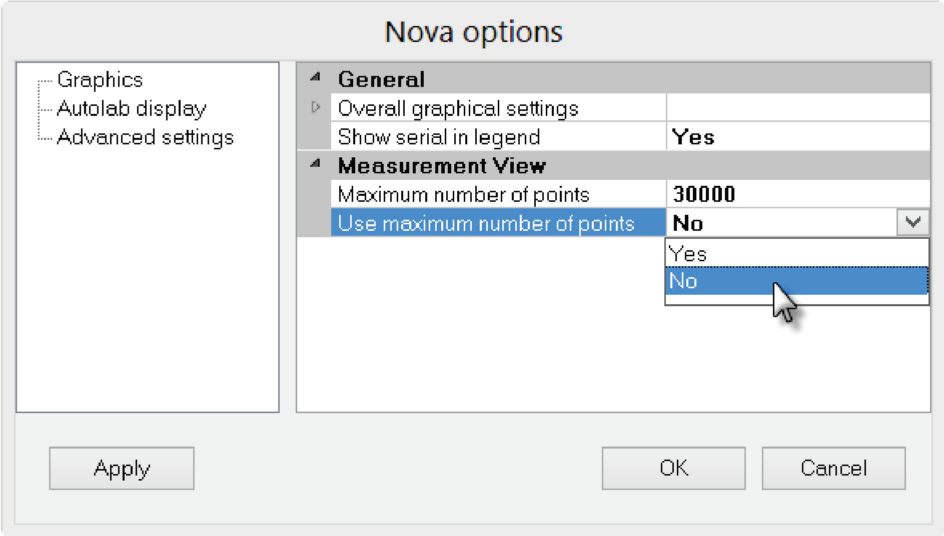


Figure 1.48 – It is possible to overrule the Maximum number of points setting

When the *Use maximum number of points* property is set to No, the software will try to display all the measured data points in each plot area (up to a limit of 10000 data points for each individual plot).

1.6.4.2 – Autolab display

In this category, the colors used in the Autolab display can be defined (see Figure 1.49).

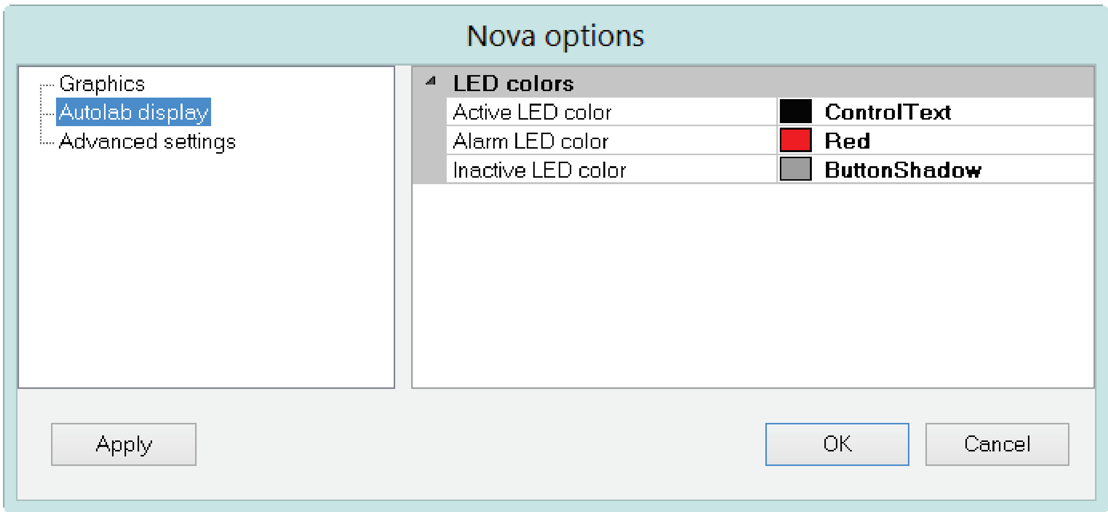


Figure 1.49 – The colors used in the Autolab display can be changed

Three different colors are used in the Autolab display. The Active LED color is used to highlight active settings of the instrument and indicate the noise levels for potential and current signals in the Autolab display. The Inactive LED is used to show the inactive settings and the Alarm LED color is used to display warnings (see Figure 1.50).

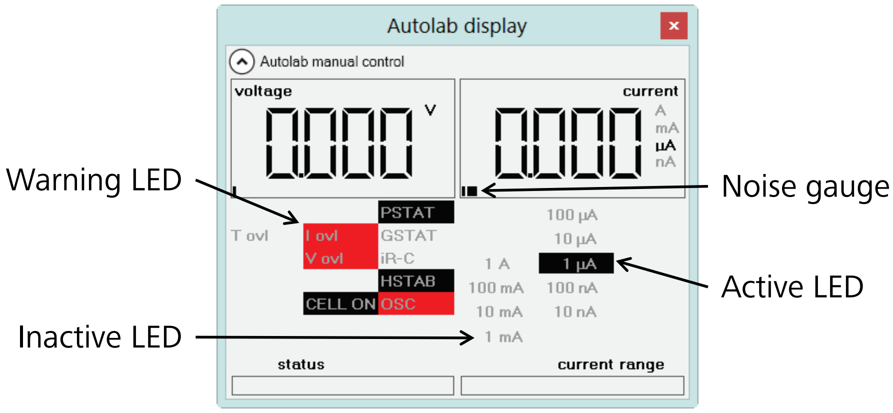


Figure 1.50 – The colors used in the Autolab display are defined in the NOVA options

1.6.4.3 – Advanced settings

In this category, advanced settings used in the software can be defined (see Figure 1.51).

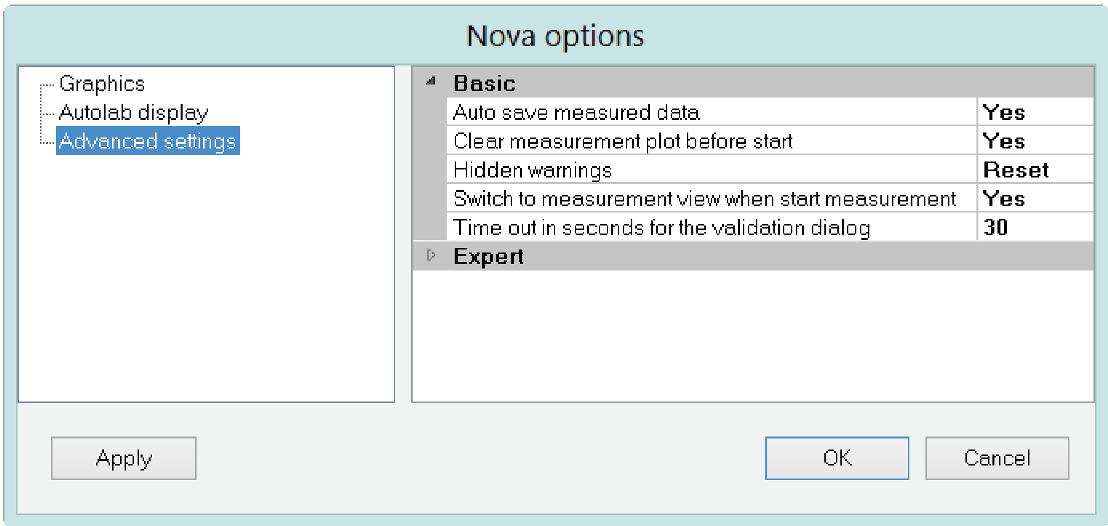


Figure 1.51 – The advanced settings of NOVA

Note

The settings are grouped into two categories: Basic and Expert. The Expert settings are used for debugging purposes and these settings should not be changed. Detailed information on these settings falls outside of the scope of this manual.

The following **Basic** settings can be changed:

- **Auto save measured data (Yes/No):** this setting defines if the data is saved automatically at the end of a measurement (default: yes). When this option is set to No, the user is prompted to specify the Name and the

Remarks for the measurement. The option to discard the data is also provided (see Figure 1.52).

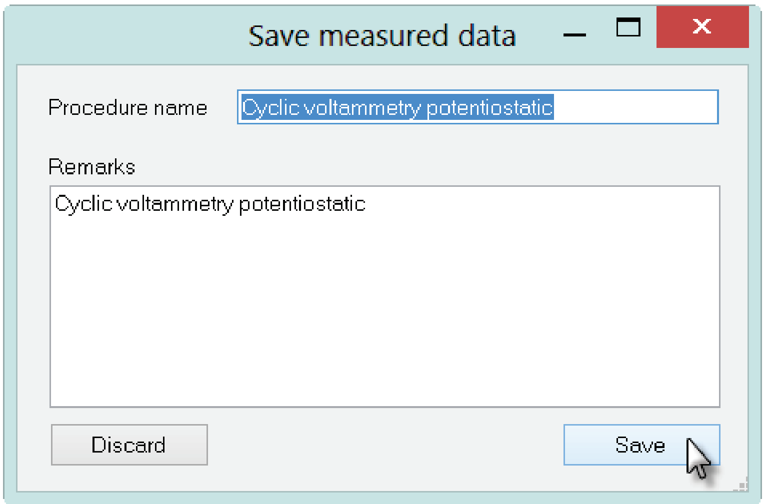


Figure 1.52 – The Save measured data dialog

- **Clear measurement plot before start (Yes/No):** this setting defines if the measurement must be cleared before each measurement (default: yes).
- **Hidden warning:** this option can be used to reset hidden warnings. Click the ... button to reset all hidden warning in the validation dialog (see Figure 1.53).

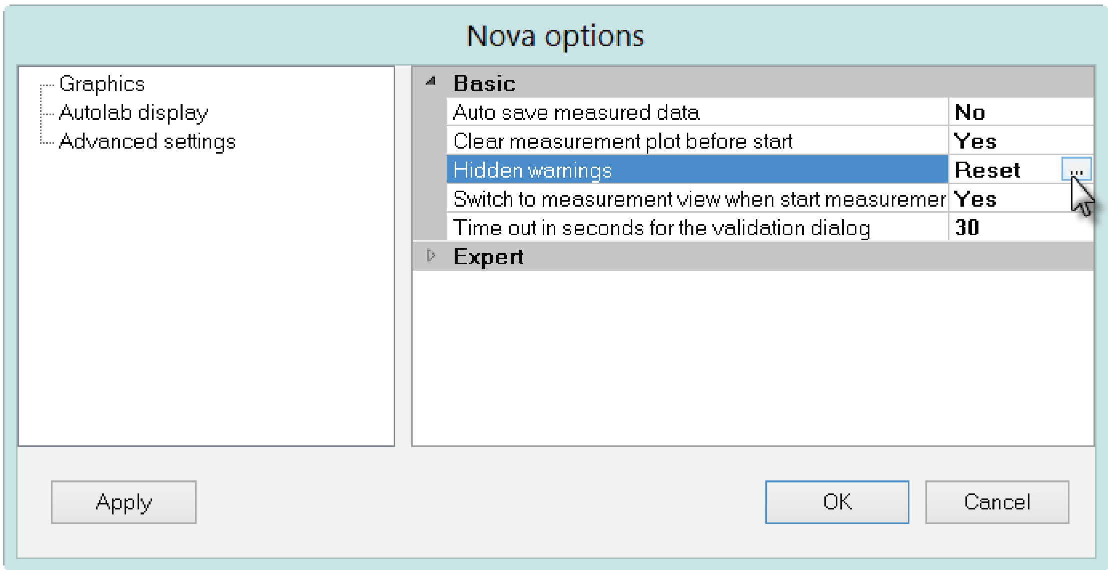


Figure 1.53 – Click the ... button to reset the hidden warnings

Warnings can be hidden in the validation screen by using the right-click menu (see Figure 1.54).

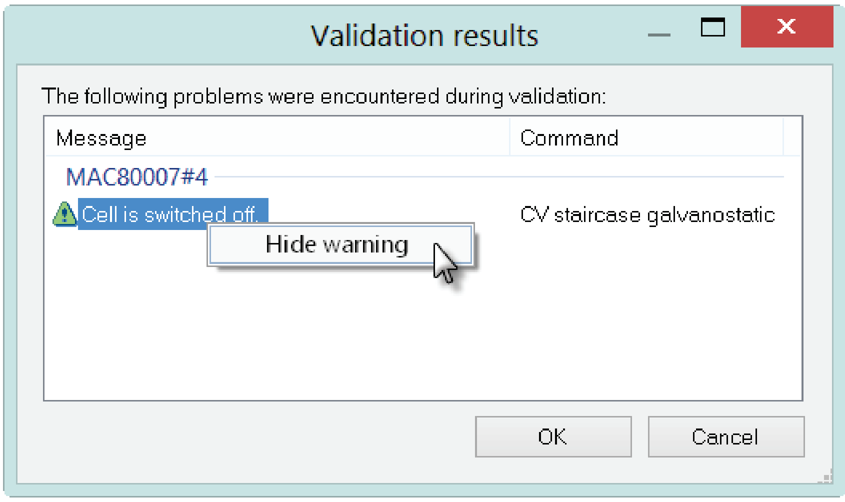


Figure 1.54 – Hiding a warning in the validation screen

- **Switch to measurement view when start measurement (Yes/No):** this setting defines if NOVA should switch to the measurement view when a measurement starts (default: yes).
- **Time out in seconds for validation dialog:** this value is the time out used when a Warning message is displayed during procedure validation (see Figure 1.55). The default value is 30 seconds.

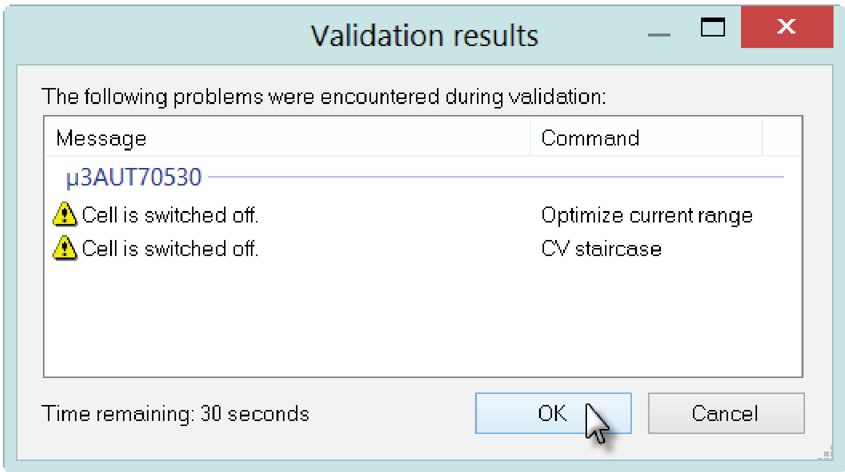


Figure 1.55 – A time out is used when a Warning is displayed in the Procedure validation

2 – The Setup view

Procedure setup is usually the first and most important step in an electrochemical measurement. This critical part of the experiment must be carefully prepared because mishaps during the measurement of the experimental data can have unpleasant consequences. More often than not, these problems can be attributed to poor experiment design.

This chapter will focus on procedure editing and will cover all the aspects of designing an electrochemical experiment using NOVA. Some examples will be provided along the way.

2.1 – The workspace

Setting up a procedure is done using the **setup view**. This view can be selected by clicking the corresponding button in the toolbar or by selecting the Setup View from the View menu (see Figure 2.1).

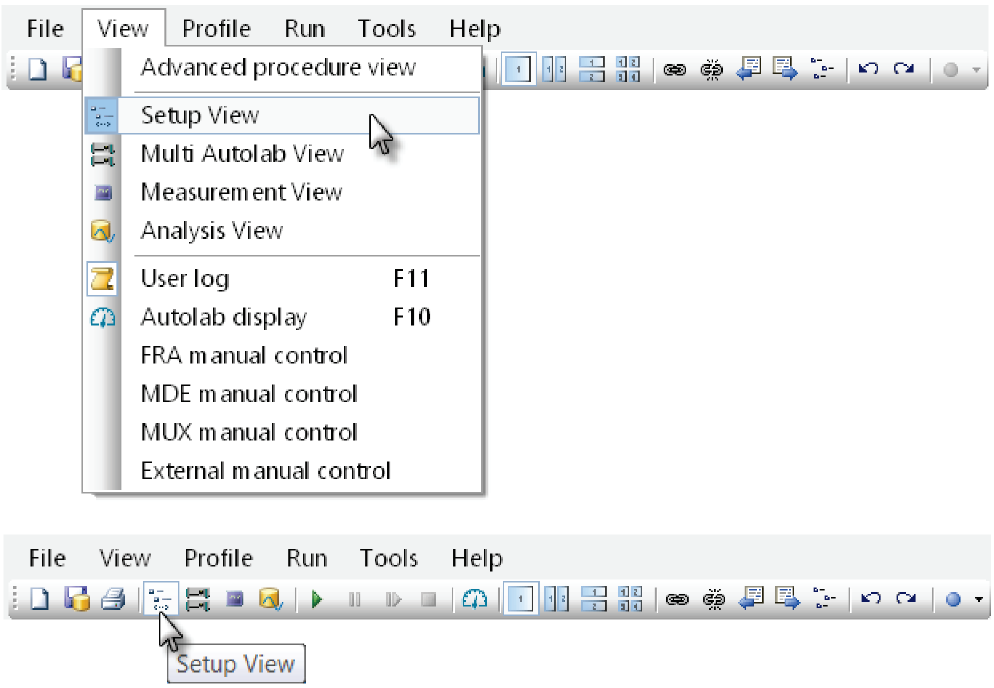


Figure 2.1 – Selecting the Setup view from the toolbar or the View menu

The setup view, which is the default startup view of NOVA, has several important features shown in Figure 2.2.

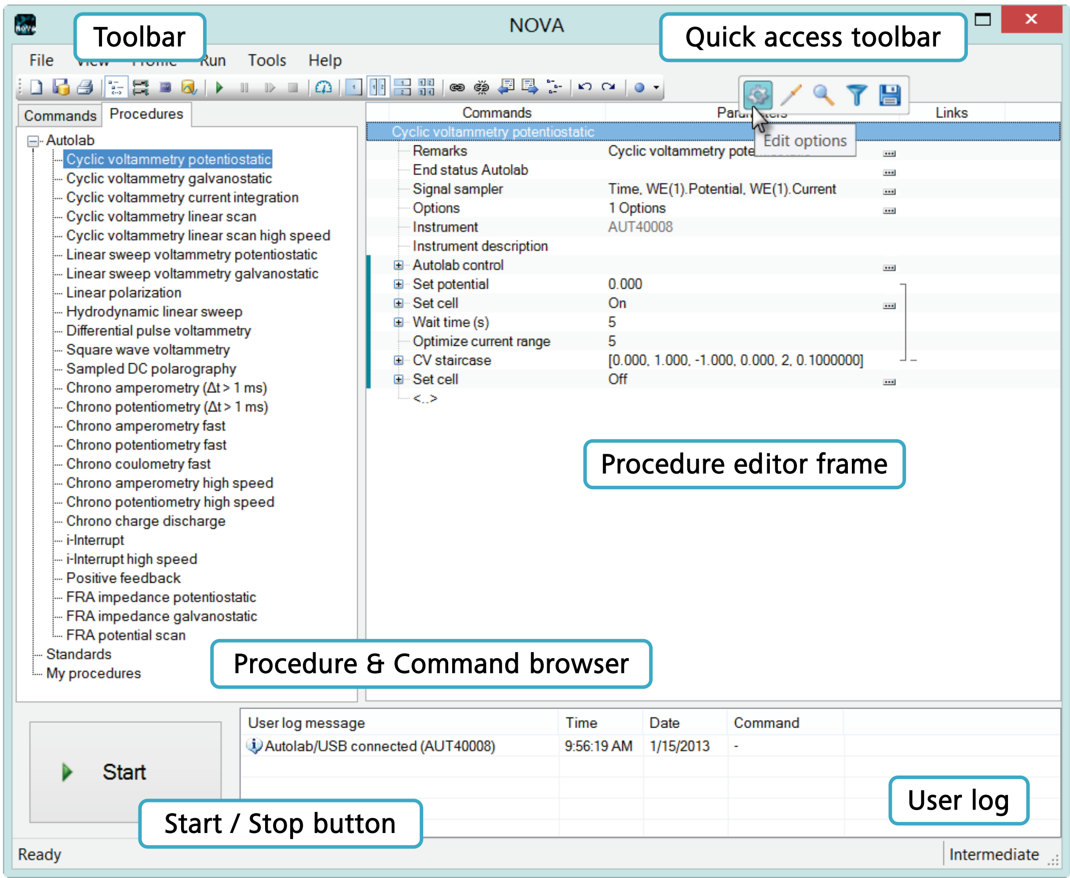


Figure 2.2 – Overview of the Setup view

Note

The User log and the Toolbar are elements common to all the views of the Nova software. The Start button is visible in all the views, except the Multi Autolab view.

The two most important parts of the Setup view are the **procedure editor frame**, which provides a visual workspace for creating/editing procedures and the **procedure and command browser frame**, on the left of the screen, which displays a list of available procedures and commands. The toolbar also features some procedure editing related buttons that will be described in this chapter.

2.2 – The procedure editor and the structure of procedures

The procedure editor provides a framework for editing procedures. An existing procedure can be loaded into this frame from the procedure browser and can then be edited to fit your needs.

When NOVA is started for the first time, the procedure editor frame is empty (Seven lines are displayed: New procedure, Remarks, End status Autolab, Signal sampler, Options, Instrument and Instrument description) and the procedure browser frame only lists procedures under the Autolab group (see Figure 2.3).

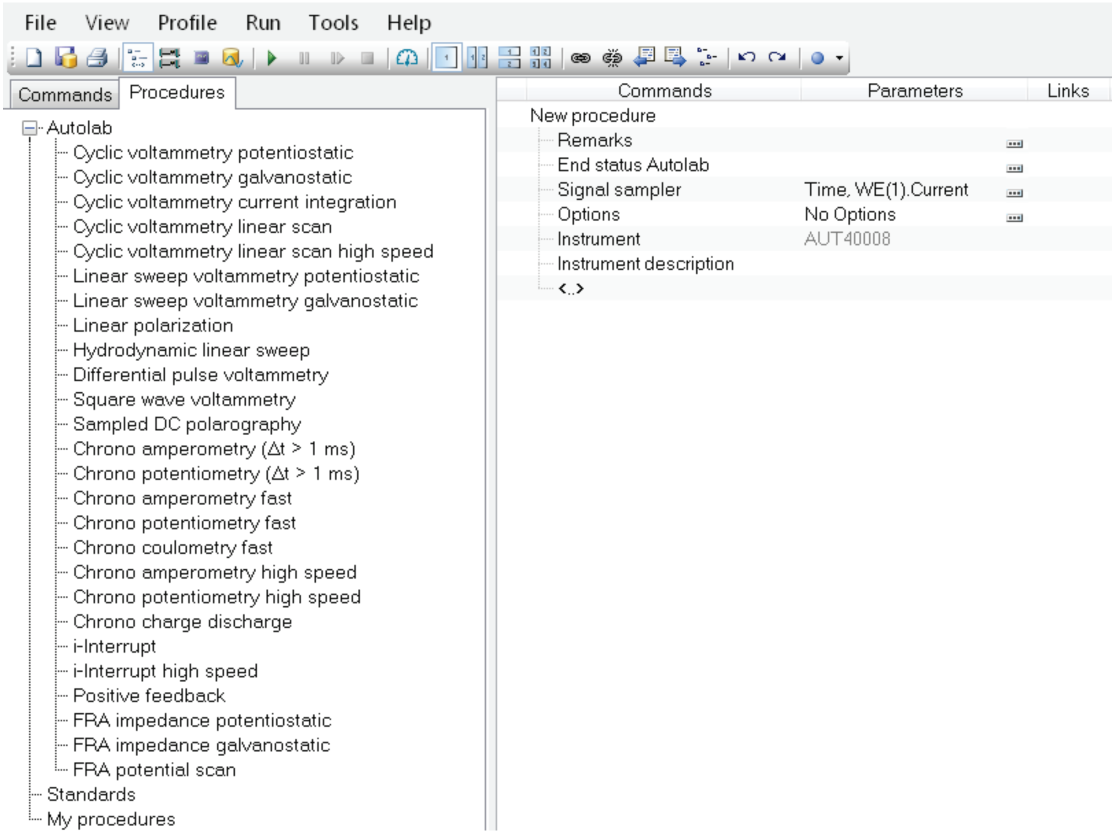


Figure 2.3 – The empty procedure editor frame and the factory standard procedures located in the Autolab group

An *empty* procedure editor frame always displays the seven lines shown in Figure 2.3 and these can be edited during the procedure creation process. *New procedure* is the default name for all new procedures.

The procedures displayed under the Autolab group in the procedure browser frame are factory standard procedures. They are always available in NOVA and cannot be deleted.

To load a procedure into the editor frame, right-click an existing procedure in the procedure browser frame and select the **Open for editing** option from the menu (see Figure 2.4). It is also possible to double click the procedure to load it in the procedure editor.

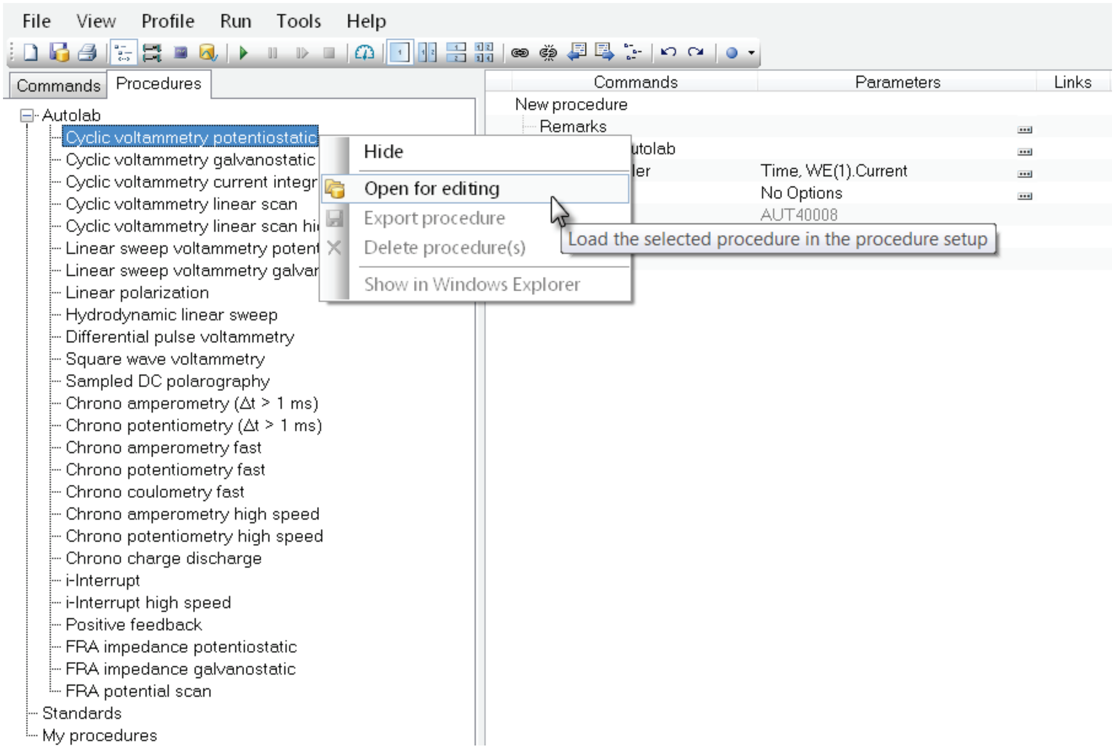


Figure 2.4 – Loading the cyclic voltammetry potentiostatic procedure into the procedure editor frame

When the cyclic voltammetry potentiostatic procedure is loaded into the procedure editor, the contents of the procedure are displayed (see Figure 2.5).

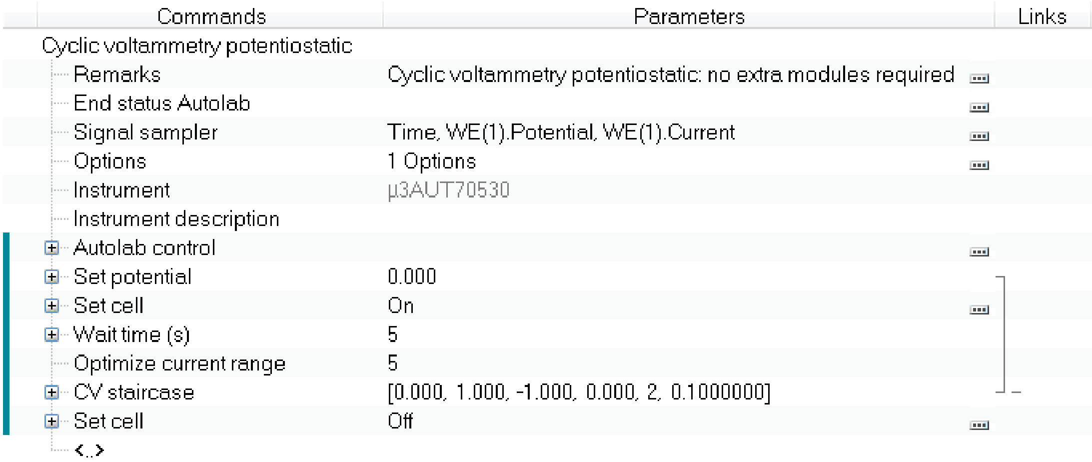


Figure 2.5 – The procedure editor frame with the loaded cyclic voltammetry potentiostatic procedure

The cyclic voltammetry potentiostatic procedure is now displayed in the procedure editor frame.

The measurement sequence in this procedure contains a series of commands. The sequence of commands defines the sequence of event that need to take place during the electrochemical measurement. The commands are executed sequentially.

Click the button to expand the details of the *CV staircase* command, as shown in Figure 2.6. It is possible to repeat this for all the commands in the measurement.

Note

It is possible to click the *Show all levels* button in the toolbar to expand the displayed procedure in order to reveal all the details of the procedure parameters.

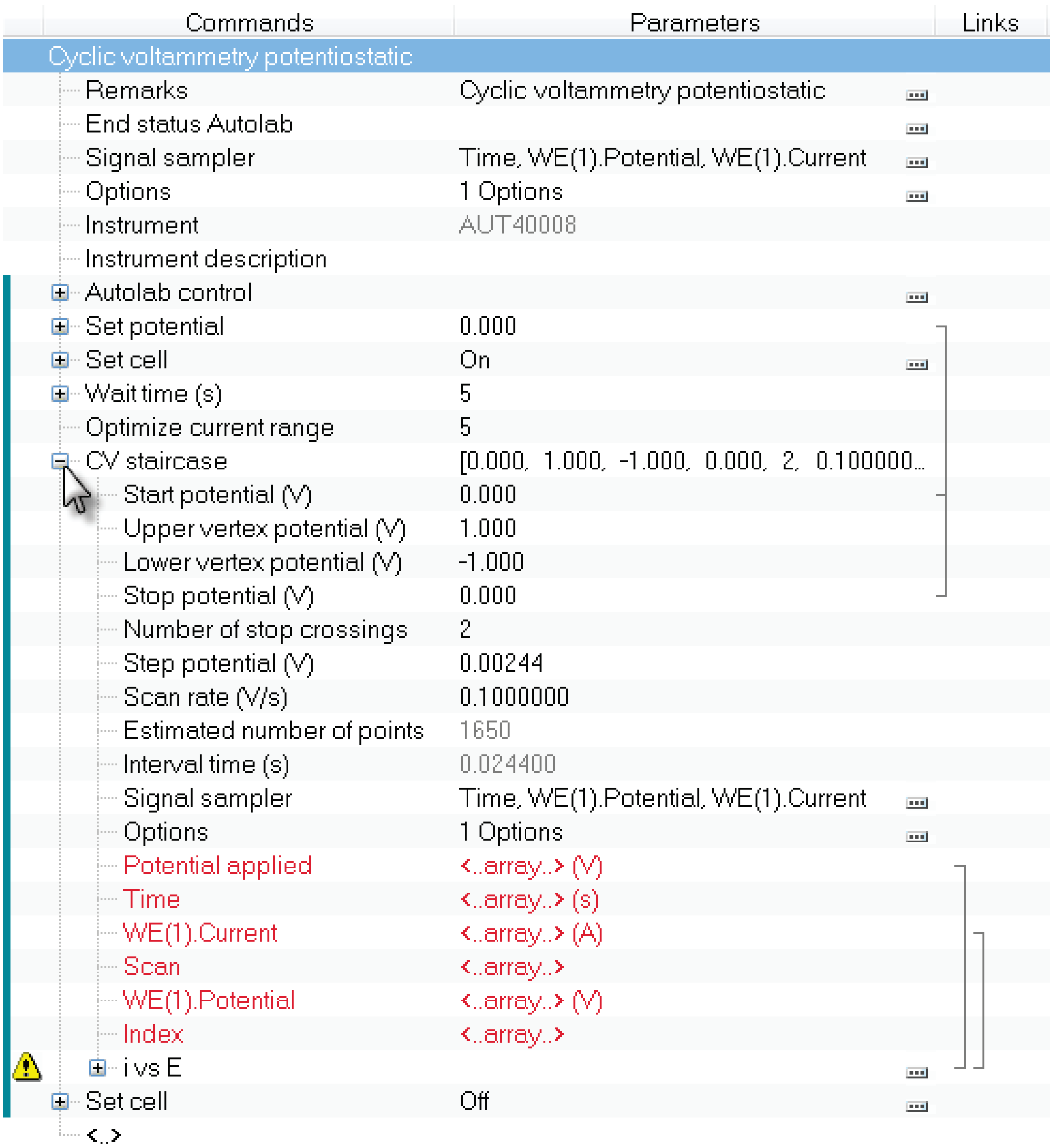


Figure 2.6 – Revealing the parameters of the cyclic voltammetry procedure and the general structure of a NOVA procedure. The highlighted red lines correspond to the electrochemical signals sampled during the measurement

A NOVA procedure has a number of components, shown in Figure 2.6.

- **Title:** this is the user-defined title of the procedure, which is located on top of the procedure editor.
- **Remarks:** this field is used to add a general comment to the procedure, like experimental conditions, reference electrode used, temperature, concentration of the supporting electrolyte, etc.
- **End status Autolab:** this instruction provides the settings of the instrument which are to be used when the measurement is finished or if the measurement is aborted by the user or by a cutoff condition.
- **Signal sampler:** this part of the procedure defines the electrochemical signals that are sampled during the measurement as well as the timing of the sampling of these signals in the course of the experiment. A more detailed description of the signal sampler is provided in Section 1.3.3 of this manual. The Autolab cyclic voltammetry potentiostatic procedure has the following signals defined in its sampler:

    - Potential applied
    - Time
    - WE(1).Current
    - Scan
    - WE(1).Potential
    - Index

- **Option(s):** this field contains optional instructions to be used during the measurement. More information on the options can be found in Section 1.3.4 of this manual. In the case of the Autolab Cyclic voltammetry potentiostatic procedure, the defined option(s) field authorizes the Autolab to use the automatic current ranging (1 mA to 100 nA).
- **Instrument:** this field is automatically filled by the software, displaying the identifying serial number of the active instrument.
- **Instrument description:** this field can be used to assign a small description to the active instrument.
- **Procedure:** consists of a series of **Commands** (listed under the Commands column) and a series of **Parameters** (listed under the Parameters column).
- **Timing guide:** this is a visual indicator of the timing accuracy of the sequence of commands. The timing is displayed using a green line on the left-hand side of the procedure editor. Interruptions in the green line indicate that the timing of the sequence will be interrupted.
- **Link:** the lines drawn on the right hand side of the procedure editor frame are an *optional* links between two or more parameters (listed under the Links column). In the case of the Autolab cyclic voltammetry potentiostatic procedure, for example, a link is used to link the *Set potential* command with the Start potential and the Stop potential from the *CV staircase* command. Links are a critical part of the NOVA procedures and they are discussed in much more detail in Section 2.4.8 of this Chapter.

- **Validation symbols:** the parameters and commands are validated in real time. When errors or warning situations are identified, a suitable symbol is provided on the left-hand side of the procedure editor to provide a visual indicator about an error or a warning.

2.3 – Simple editing

The easiest form of procedure editing in NOVA consists of loading a pre-defined procedure into the procedure editor, modifying some of the parameters of the procedure and running the experiment.

This very simple mode of operation will be explored in this section of the manual. The next sections will describe advanced procedure editing.

The default Autolab cyclic voltammetry potentiostatic procedure is designed to run one staircase cyclic voltammogram, starting from a potential of 0 V, going up to a potential of 1 V, then back to -1 V and finally stopping again at 0 V. The scan rate is 100 mV/s and the step potential is 2.44 mV.

These experimental parameters can be found in the expanded view of the procedure, shown in more detail in Figure 2.7.

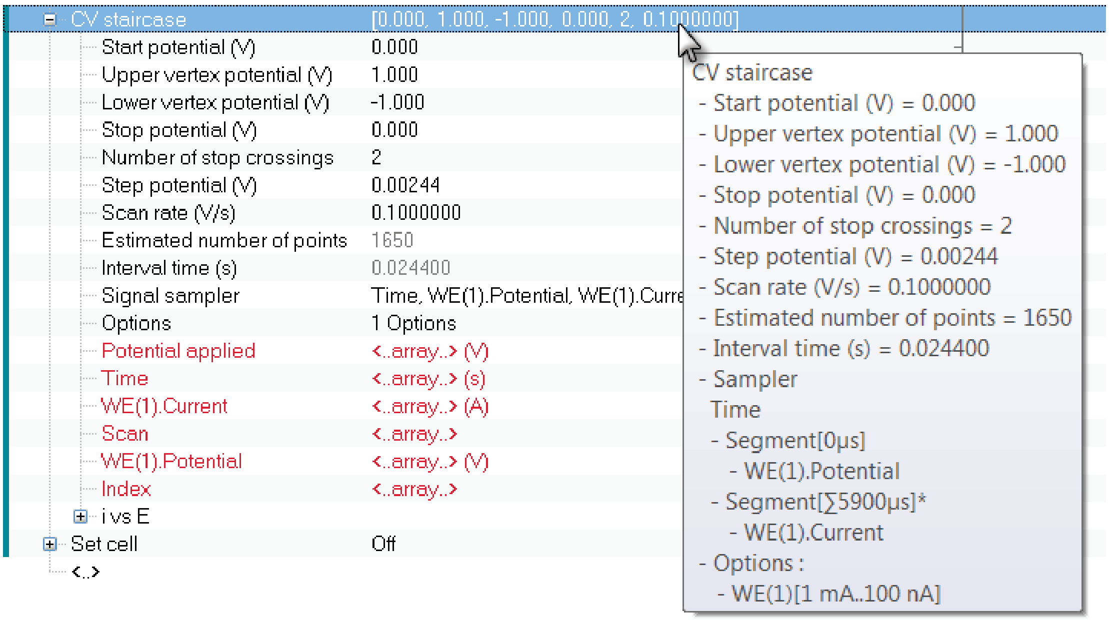


Figure 2.7 – A detailed view of the cyclic voltammetry potentiostatic procedure and its default parameters (note the detailed tooltip)

Each command shown in the expanded view of the procedure has a number of parameters that can be edited. These parameters are indicated in **black** (Start potential, Scan rate, etc). Some values are indicated in light grey. This means that they cannot be edited by the user but that they are dependent on the other user-defined parameters. Figure 2.7 shows that the *CV staircase* command has seven parameters that can be edited by the user as well as a signal sampler and options.

The six lines shown in red are the electrochemical signals that are sampled during the measurement.

Each command shows a summary of its parameters between brackets. This is quite useful because it provides an overview of the parameters of the command even if it is displayed in its contracted form (see Figure 2.8).

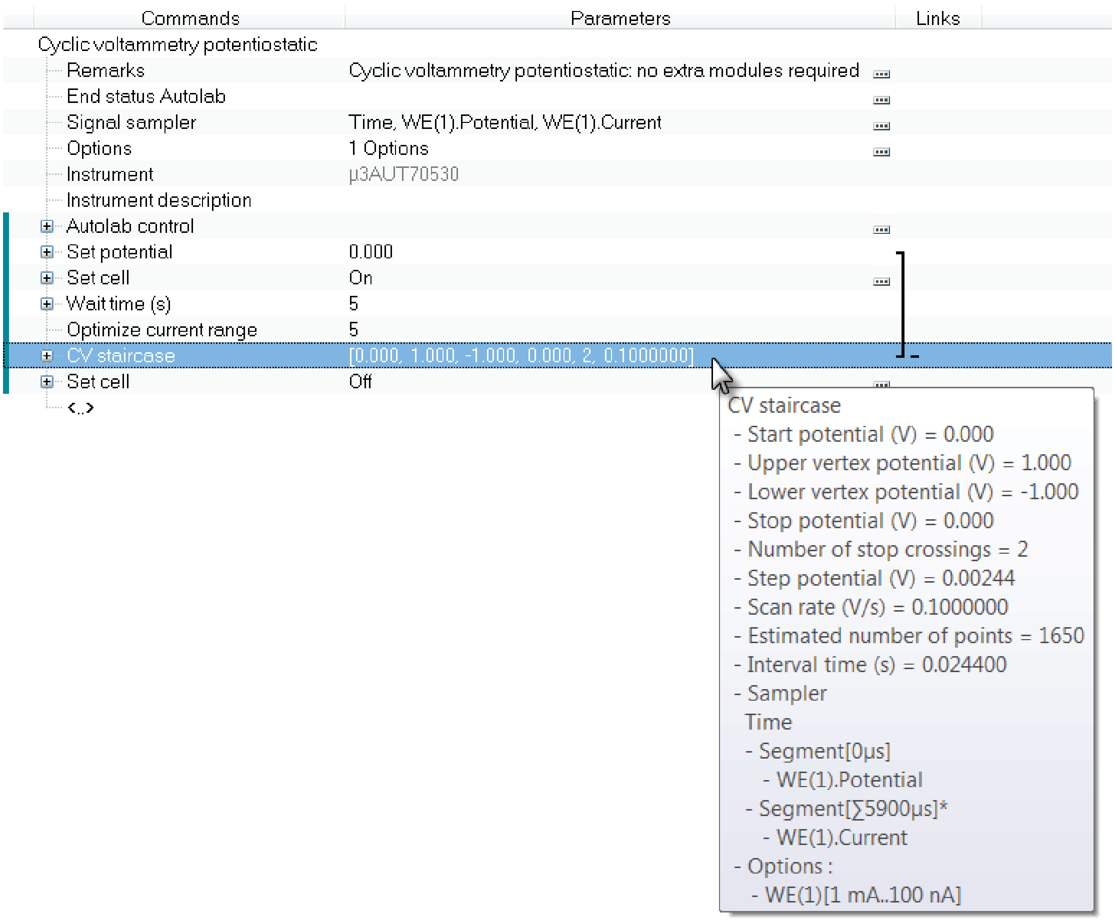


Figure 2.8 – A detailed view of the *CV staircase* command in the contracted form

> **Note**
>
> Positioning the mouse pointer on the *CV staircase* command will display a tooltip which provides an overview of the parameters (see Figure 2.7 and Figure 2.8).

The five commands preceding the *CV staircase* command in this procedure (*Autolab control*, *Set potential*, *Set cell*, *Wait time* and *Optimize current range*) are used as a pre-treatment.

To change a parameter, expand the *CV staircase* command and click the parameter you want to edit. Type in the new value of the parameter and press the enter key to confirm the change. Figure 2.9 shows a modification of the scan rate of the *CV staircase* command from 0.1 V/s to 0.2 V/s. A tooltip reminding you to enter the scan rate in V/s is displayed.

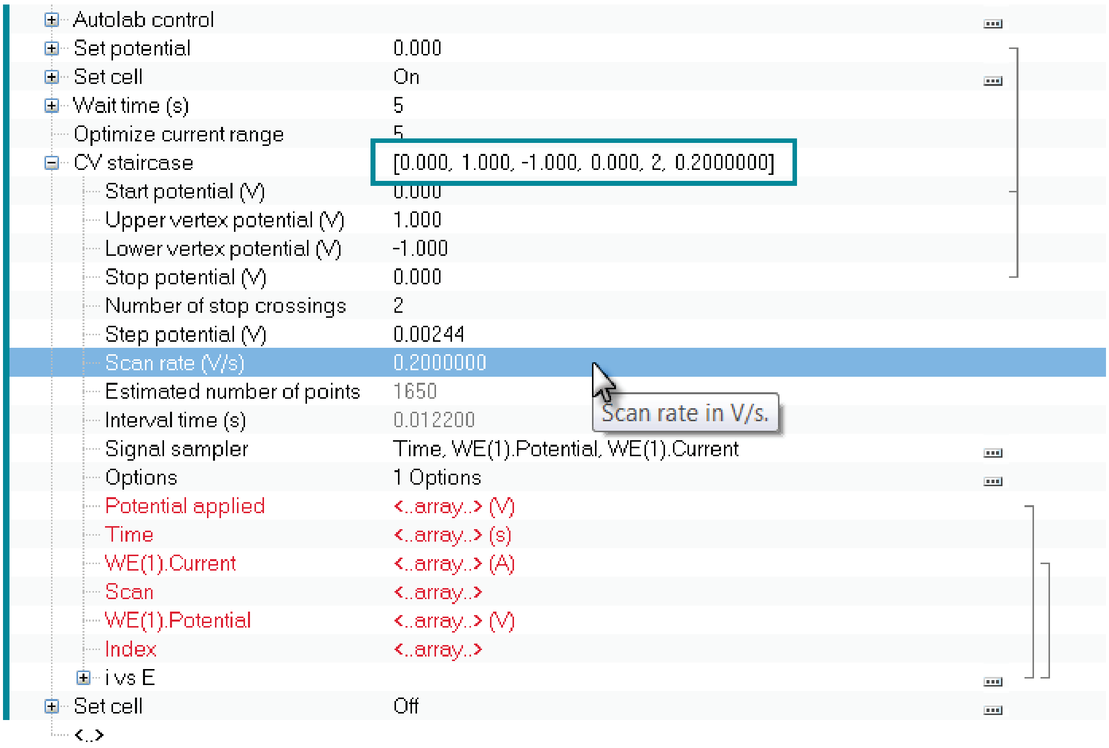


Figure 2.9 – Modifying the scan rate

Note

When the scan rate is changed, the line shown between brackets next to the CV Staircase command is updated (from [0.000, 1.000, -1.000, 0.000, 2, 0.10000] to [0.000, 1.000, -1.000, 0.000, 2, **0.20000**]).

Changing the scan rate of the cyclic voltammetry procedure has an effect on some of the grey parameters. The interval time originally was 24.4 ms and now is 12.2 ms, because the scan rate has been doubled. If the upper vertex potential is changed to 0.8 V, the Estimated number of points will also change (see Figure 2.10).

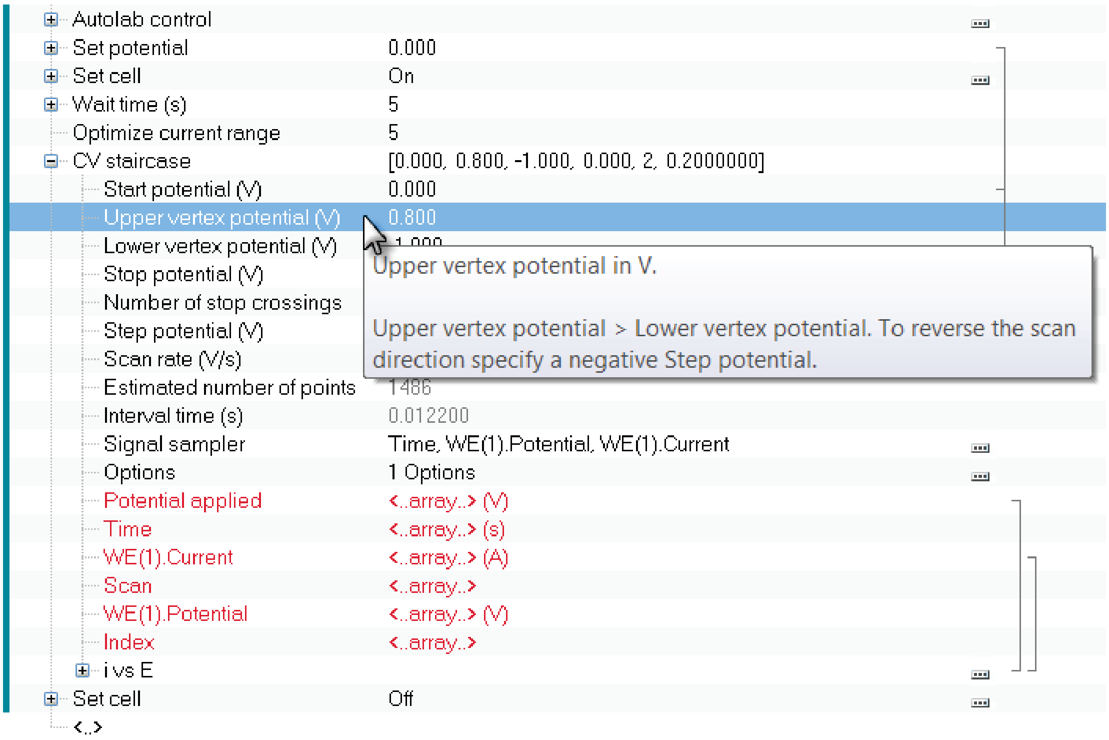


Figure 2.10 – Changing the upper vertex potential has an effect on the Estimated number of points

The procedure can now be started using the new experimental parameters.

2.4 – Advanced editing

The previous section described a very straightforward use of the NOVA procedure editor: loading a procedure, editing some parameters and running the experiment.

This section will cover a more advanced use of the procedure editor. It is the most important part of this chapter. Special attention will be given to concepts like **timing** and **linking**.

2.4.1 – Adding/Removing commands

Section 2.3 described the use of ready-made Autolab procedures. While these procedures can be used without modifying their structure, it might be necessary to change them to fit a specific need. A typical example is the standard **Chrono amperometry** procedure.

Load the Chrono amperometry (Δt > 1 ms) procedure into the procedure editor. This procedure has a pre-treatment step just like in the case of the cyclic voltammetry procedure. During this procedure, the potential of each step is set using the *Set potential* command and the response of the cell is measured using the *Record signals (>1 ms)* command.

In the standard Autolab Chrono amperometry (Δt > 1 ms) procedure, three potential steps are applied using the *Set potential* command, and the response of the cell is recorded for each step using the *Record signals (>1 ms)* command. The duration is set to of 5 seconds and an interval time of 0.01 seconds is used. The first step is at 0 V (the pre-treatment potential), then at 0.5 V and finally at -0.5 V (see Figure 2.11).

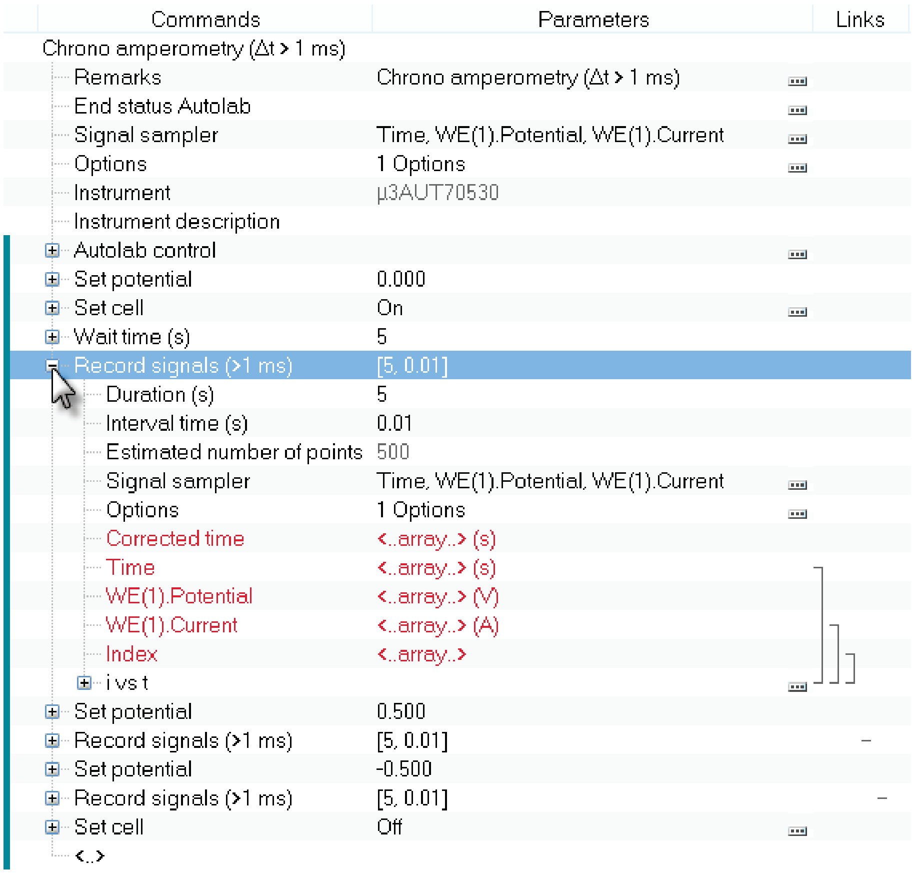


Figure 2.11 – The default Autolab Chrono amperometry (Δt > 1 ms) procedure (the *Record signal (> 1 ms)* command is expanded to show the details of the command)

The potential used for each step can be edited in the same way as the previous example. However, editing a chrono amperometry measurement is not only done by changing the duration of the potential steps and the interval time, but also by changing the number of potential steps.

Note

Nova has been developed according to the Windows guidelines. This means that the user interface of the software works as any other Windows application. Selection of multiple items while holding the CTRL key, drag & drop, dragging while holding the CTRL key, context-sensitive menus through the right mouse button and using the mouse wheel are all standard Windows actions, which are present in Nova.

2.4.2 – Removing commands

To remove potential steps from this procedure, both the set potential and the record signals command have to be removed. Hold the **CTRL** key on the keyboard and click the two commands for the third potential step and press the **delete** key. It is also possible to use the right-click button and choose the **Delete** option from the context menu. This will remove both commands from the procedure (see Figure 2.12 and Figure 2.13).

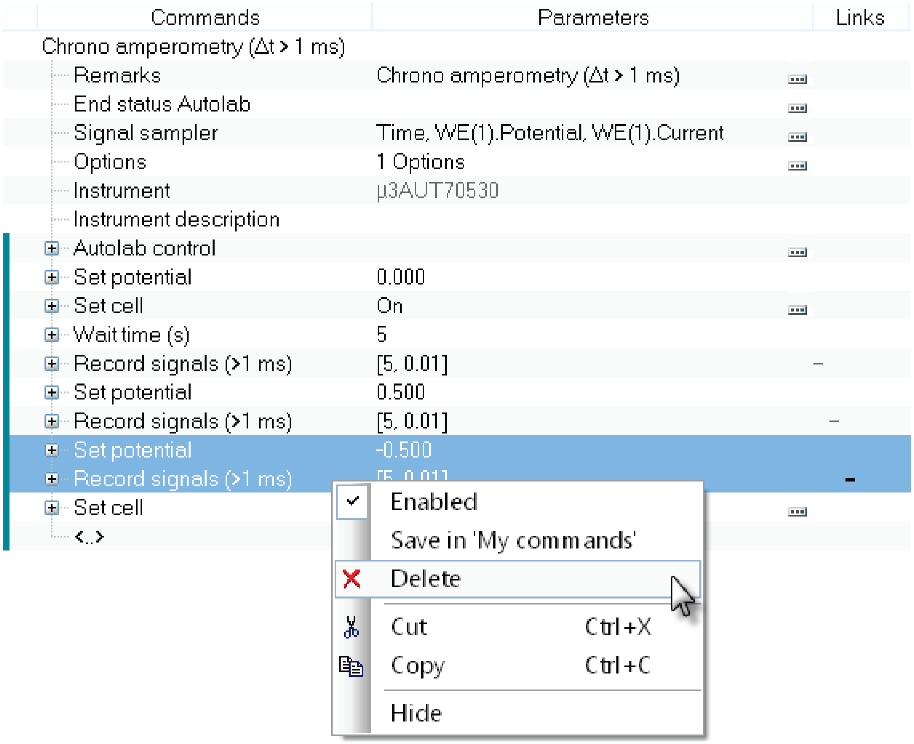


Figure 2.12 – Removing the third potential step from the procedure (1/2)

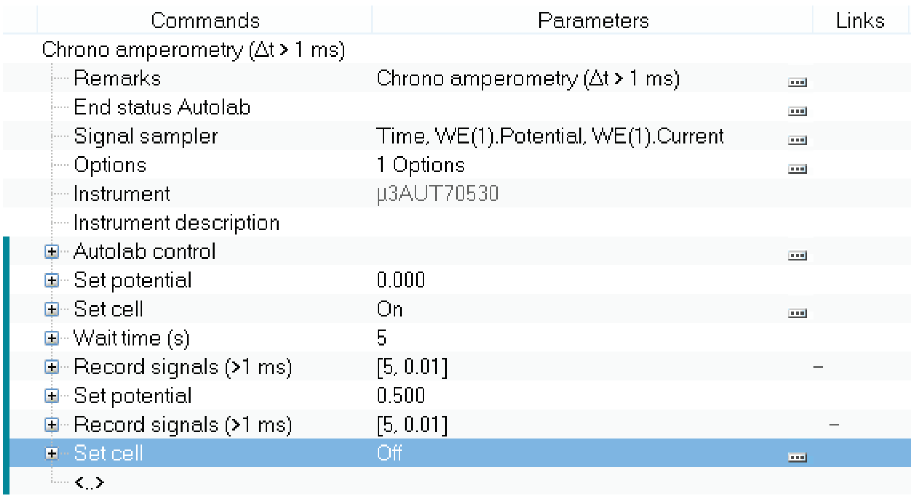


Figure 2.13 – Removing the third potential step from the procedure (2/2)

Note

It is possible to undo an action at any time by clicking the button in the toolbar or using the CTRL-Z keyboard shortcut. It is also possible to right-click the procedure editor and select the undo option from the context menu (see Figure 2.14).

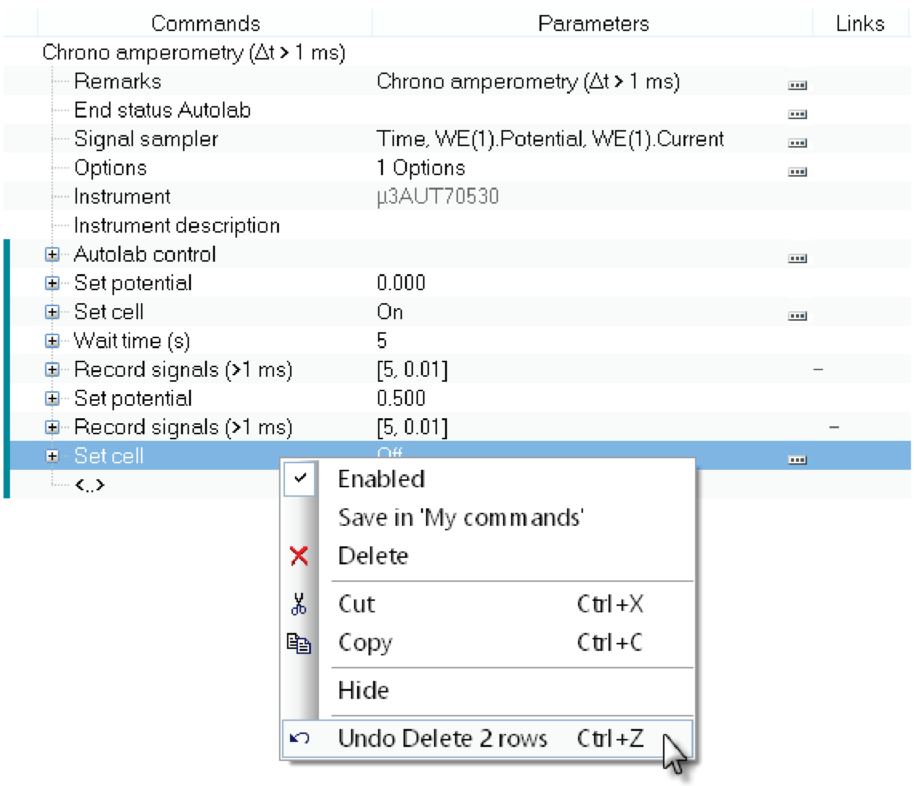


Figure 2.14 – The Undo option is also available from the right-click menu

Note

Clicking the redo button, , button in the toolbar or using the CTRL-Y keyboard shortcut will redo the last undone action. It is also possible to right-click the procedure editor and select the redo option from the context menu (see Figure 2.15).

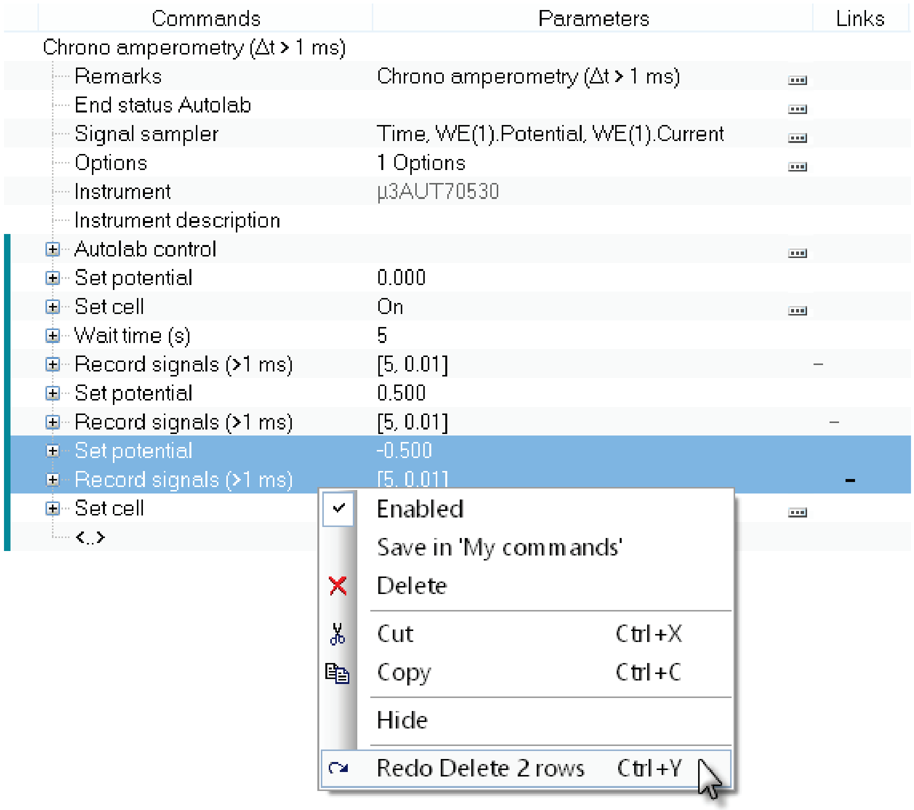


Figure 2.15 – The Redo option is also available from the right-click menu

2.4.3 – Adding commands

The procedures browser frame also has a **Commands browser**. On top of the frame, two **tabs** are located. These tabs are called *Commands* and *Procedures*, respectively. The default tab is the Procedures tab. While this tab is selected, the browser frame displays the available procedures.

If you click the Commands tab, you will switch the content of that frame to a list of commands (see Figure 2.16).

Note

Detailed information on all available commands can be found in the Command list document, available from the Help menu.

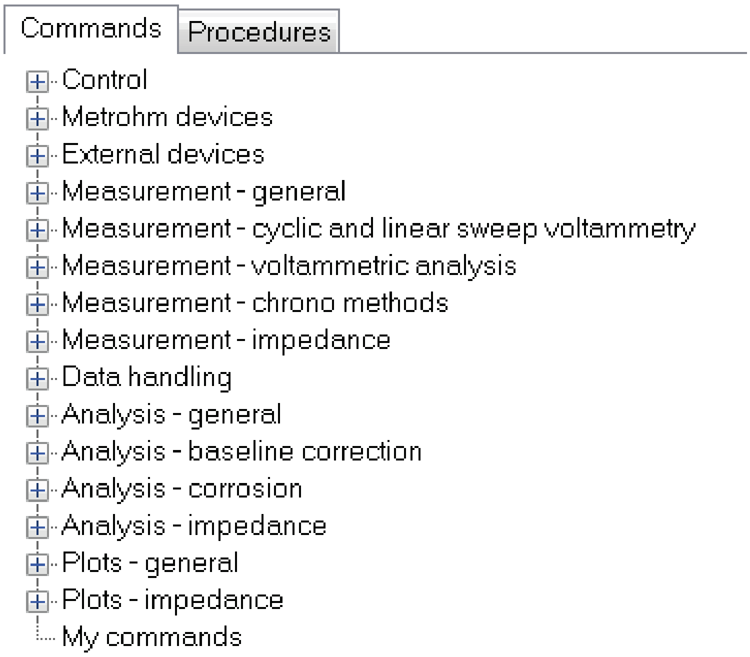


Figure 2.16 – Switching to commands browser by clicking the commands tab

The commands are grouped into sixteen categories[18].

Figure 2.17 highlights the two commands used in the chrono amperometry procedure. The *Set potential* command is located in the **Measurement – general** group. This group contains general purpose commands like Set cell, Wait time, etc. On the other hand, the *Record signals (> 1 ms)* command is located in the **Measurement – chrono methods** group. This group contains all the commands specifically related to the time resolved measurements.

Note

More information on chrono measurements can be found in the Chrono methods and the Chrono methods high speed tutorials, available from the Help menu.

[18] Please refer to the Command list description, available from the Help – Tutorials menu, for more information on each command.

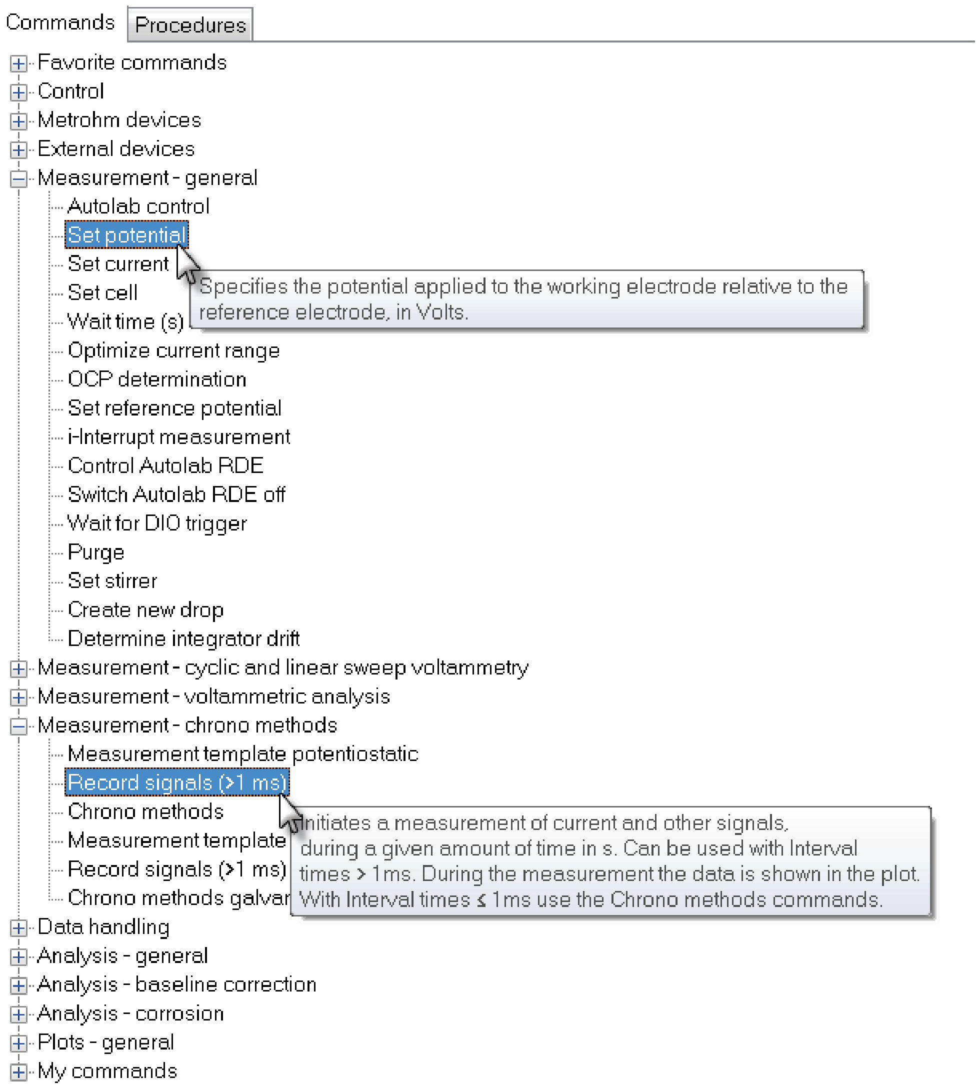


Figure 2.17 – The Set potential and *Record signals (> 1 ms)* commands used in the Chrono amperometry procedure

- The **Set potential** command is used to set a specific potential on the electrochemical cell.
- The **Record signals (>1 ms)** command is used to sample the current during a given amount of time, and using an interval time larger than 1 ms[19].

Click the *Set potential* command and, while holding the left mouse button, drag the command over to the procedure editor frame. Place the command on a convenient insertion point (a yellow arrow will appear, indicating the insertion point of the added command) and release the mouse button to confirm the insertion of the *Set potential* command (see Figure 2.18).

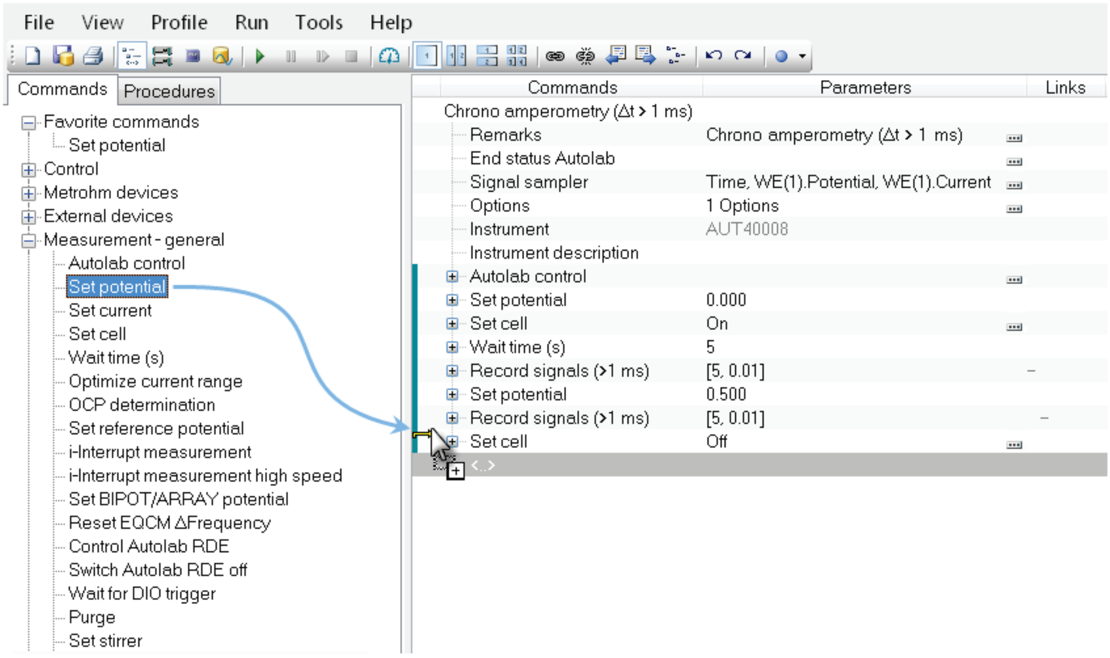
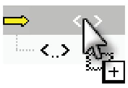
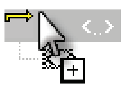


Figure 2.18 – Inserting a *Set potential* command in the procedure

Pay attention to the mouse pointer when the command is dragged on the procedure editor. Depending on the type of command and the location in the procedure editor, the mouse pointer can have three different shapes.



Add command: the command can be added at the pointer location.



Insert command: the command can be inserted at the pointer location. All commands located below this position will be shifted downwards.

Error: the pointer location is not valid for the selected command.

[19] Smallest possible value: 1.33 ms.

Note

The drag & drop action described in the last paragraph is a important part of the procedure editing process. While the final sections of this chapter will cover this topic in more detail, it is important to understand that the drag & drop action is the most common user interaction in the procedure editor environment.

Special attention has been given to building an *intelligent* drag & drop interface: Nova will only allow to drop commands in places where these commands can in fact be dropped.

Repeat the drag and drop movement to add a *Record signals (> 1 ms)* command under the newly added *Set potential* command (see Figure 2.19).

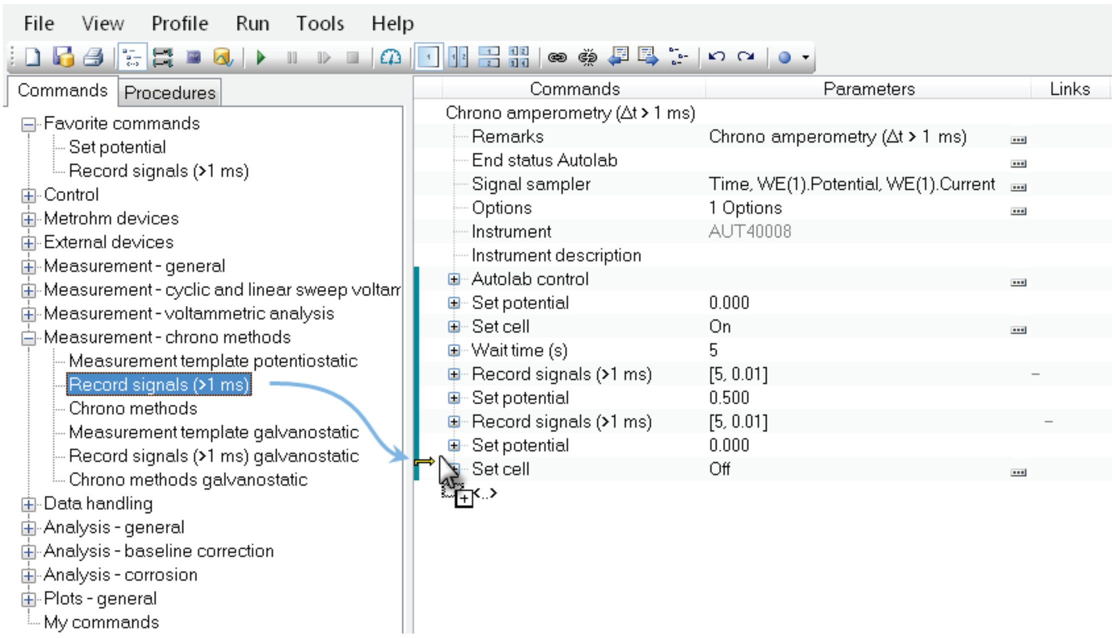


Figure 2.19 – Inserting a *Record signals (> 1 ms)* command in the procedure

Having added the two commands, their parameters can be edited in order to set the potential to the required level and sample the current for the required amount of time, with a convenient sampling rate (see Figure 2.20).

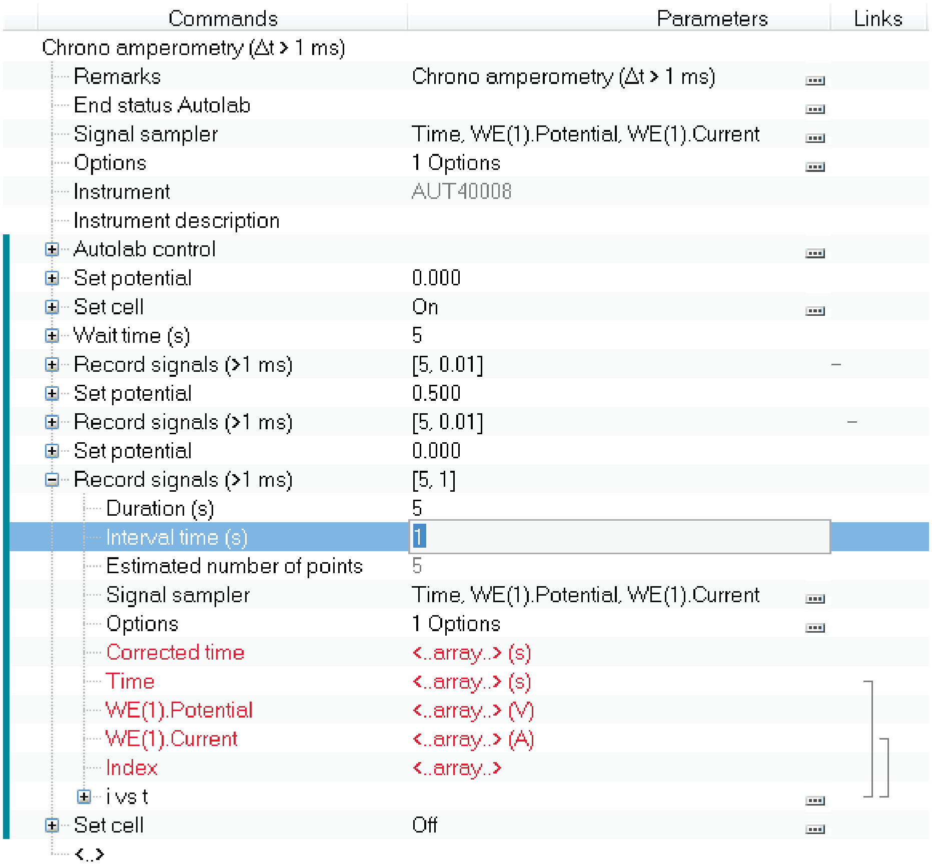


Figure 2.20 – Editing the parameters for the two added commands

Note

In the *Record signal (> 1 ms)*, the duration must be longer than the interval time.

2.4.4 – Cut and Copy – Paste commands

It is also possible to use the well known cut/copy and paste options to duplicate a command or a group of commands already present in the procedure. For example, to add an additional *Set potential* command and an additional *Record signals (> 1 ms)* command to the original Chrono amperometry (Δt > 1 ms) procedure, select the last two *Set potential* and *Record signals (> 1 ms)* and right click the selected commands. Select the Copy option from the context menu (see Figure 2.21).

Note

The CTRL-X (for Cut), CTRL-C (for Copy) and CTRL-V (for Paste) keyboard shortcuts also work.

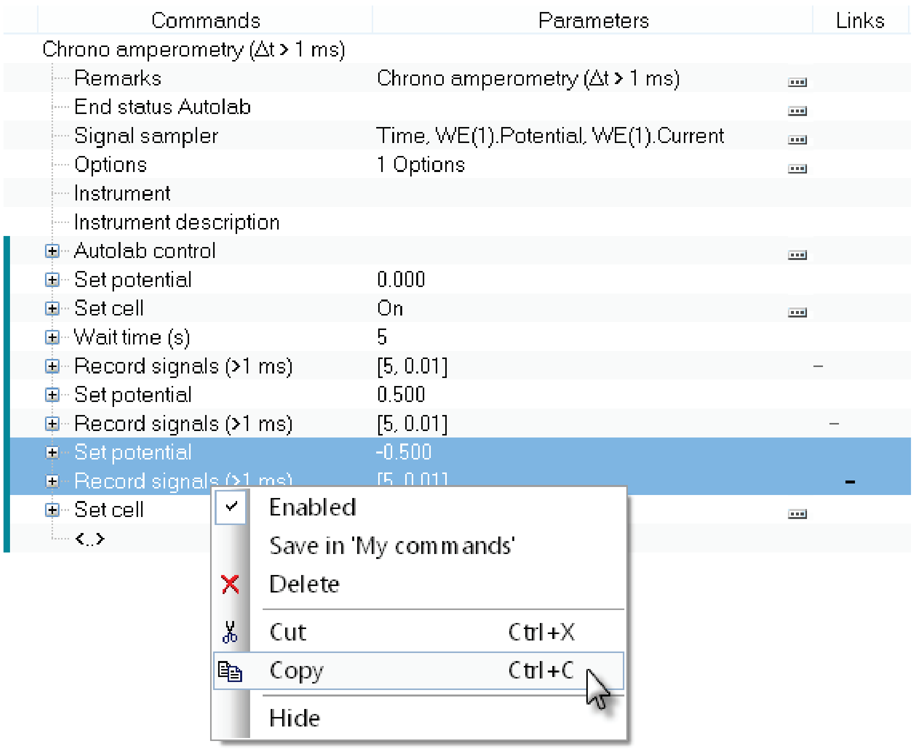


Figure 2.21 – Select the copy option from the right-click menu

To paste the copied (or cut) commands, right-click the command **below** which the commands need to be pasted and select the Paste option from the context menu (see Figure 2.22).

Note

Paste commands are always added below the selected command.

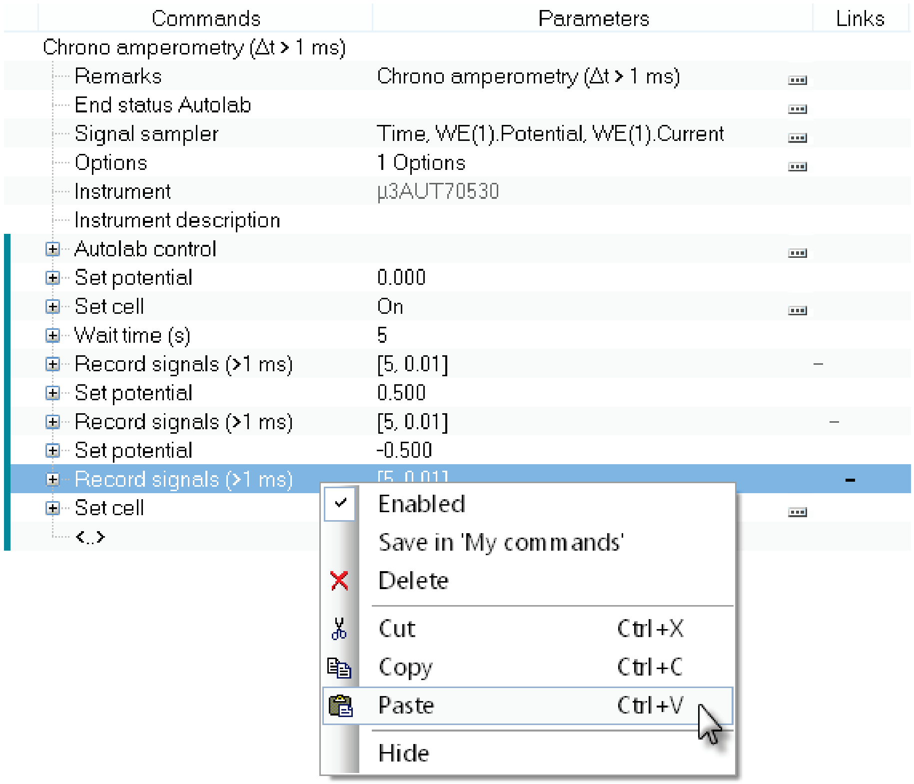


Figure 2.22 – Pasting the copied commands in the procedure

The copied commands will be added to the procedure, below the selected command (see Figure 2.23).

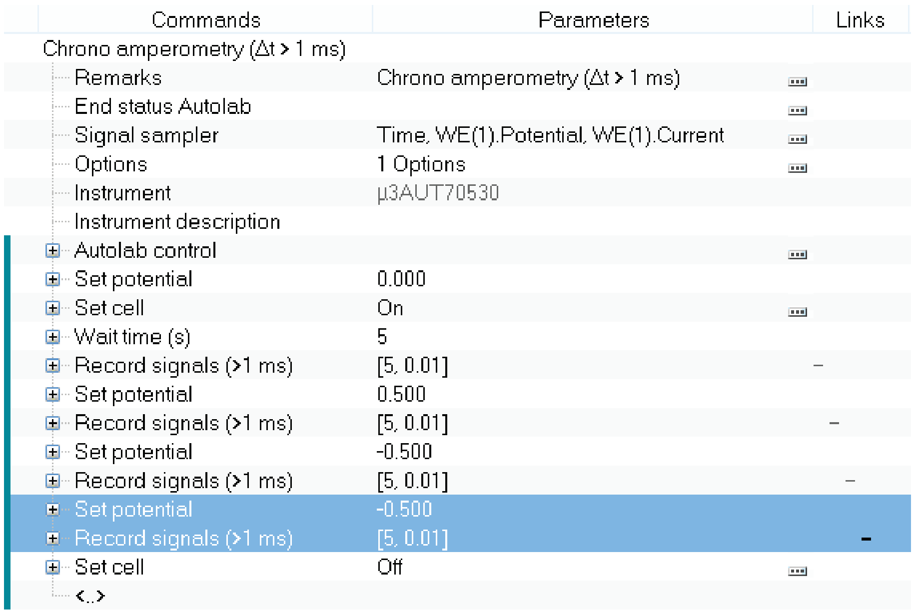


Figure 2.23 – The copied commands are added to the procedure

2.4.5 – Favorite commands

A dedicated command group, called Favorite commands, is available in the Command browser. This group, initially empty, will fill up when commands are dragged and dropped in the procedure editor, as explained in Section 2.4.3. This group will automatically keep track of the ten **most used** commands.

In Section 2.4.3, two commands have been dragged into the procedure editor: *Set potential* and *Record signals (> 1 ms)*. Since these commands have been manually added to the procedure, they are now listed in the Favorite commands group (see Figure 2.24).

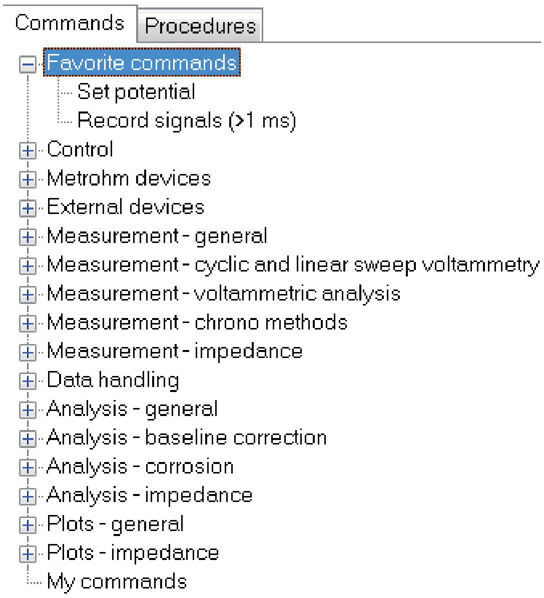


Figure 2.24 – The Favorite commands group automatically ranks the ten most used commands

The list of commands shown in the Favorite commands group is automatically updated each time a new command is manually added to the procedure. All these commands are ranked by popularity and the ten most often used commands are listed in the group at any given time. It is however possible to manually remove a command from the Favorite group by right-clicking the command and choosing the Remove from Favorite commands option (see Figure 2.25).

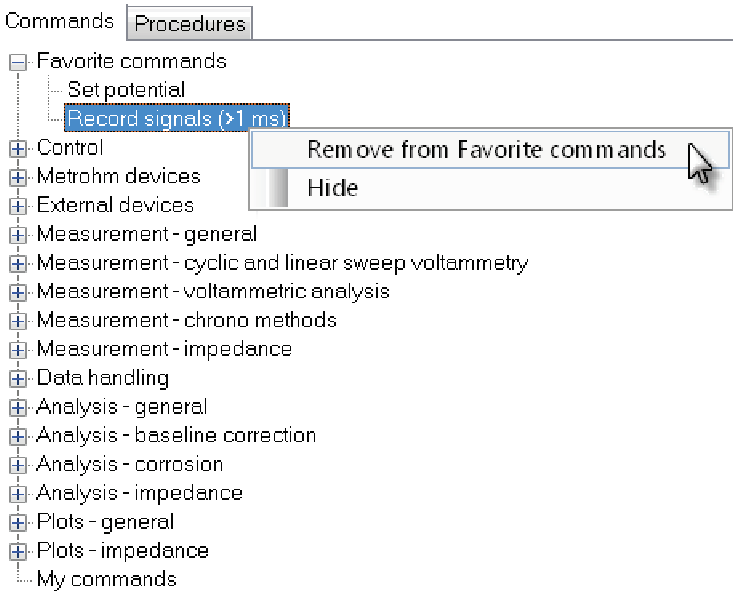


Figure 2.25 – It is possible to manually remove commands from the Favorite group using the right-click menu

2.4.6 – Enabling/Disabling commands

An alternative to removing commands from a procedure is *disabling* the commands. This is convenient because it is possible to enable these commands again later if it is necessary, without having to use the commands browser.

Reload the original Chrono amperometry procedure from the Autolab group. This procedure has three potential steps but in this example, the final potential step will be disabled.

To disable a command, right-click the command and deselect the Enabled option. In this example, disable the third Set potential command (see Figure 2.26).

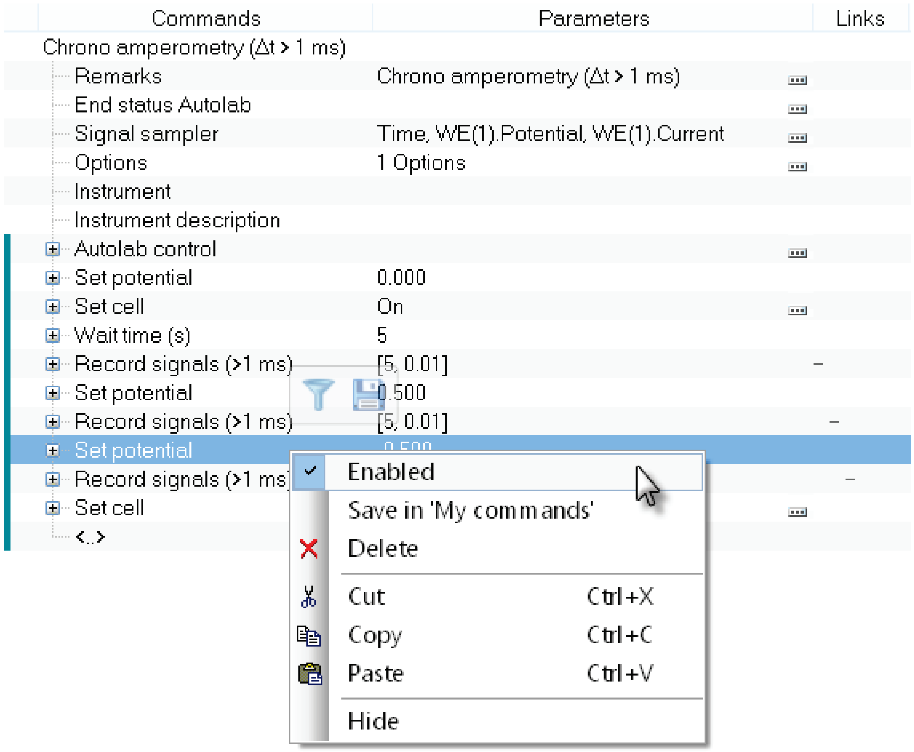


Figure 2.26 – Unchecking the Enabled option will disable the command

A disabled command will be displayed in grey in the procedure editor (see Figure 2.27).

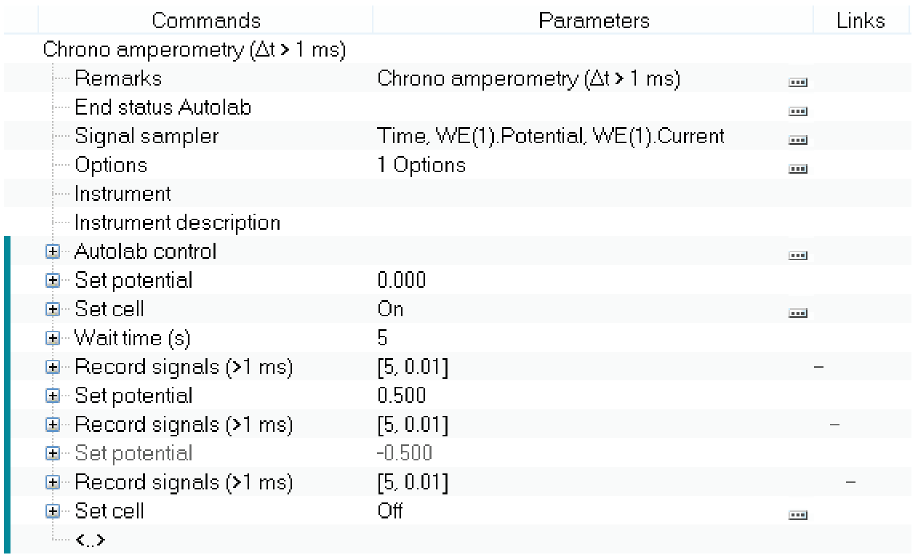


Figure 2.27 – Disabled commands are displayed in grey

If you start the experiment, you should only see two potential steps, the first step having a duration of 5 seconds and the second step having a duration of 10 seconds (5 seconds + 5 extra seconds from the third potential step, as *Record signals (> 1 ms)* command used to record the third step has not been disabled in Figure 2.27).

Figure 2.28 shows a comparison of the recorded WE(1).Potential values during the original Chrono amperometry procedure and the procedure in which the third *Set potential* command has been disabled.

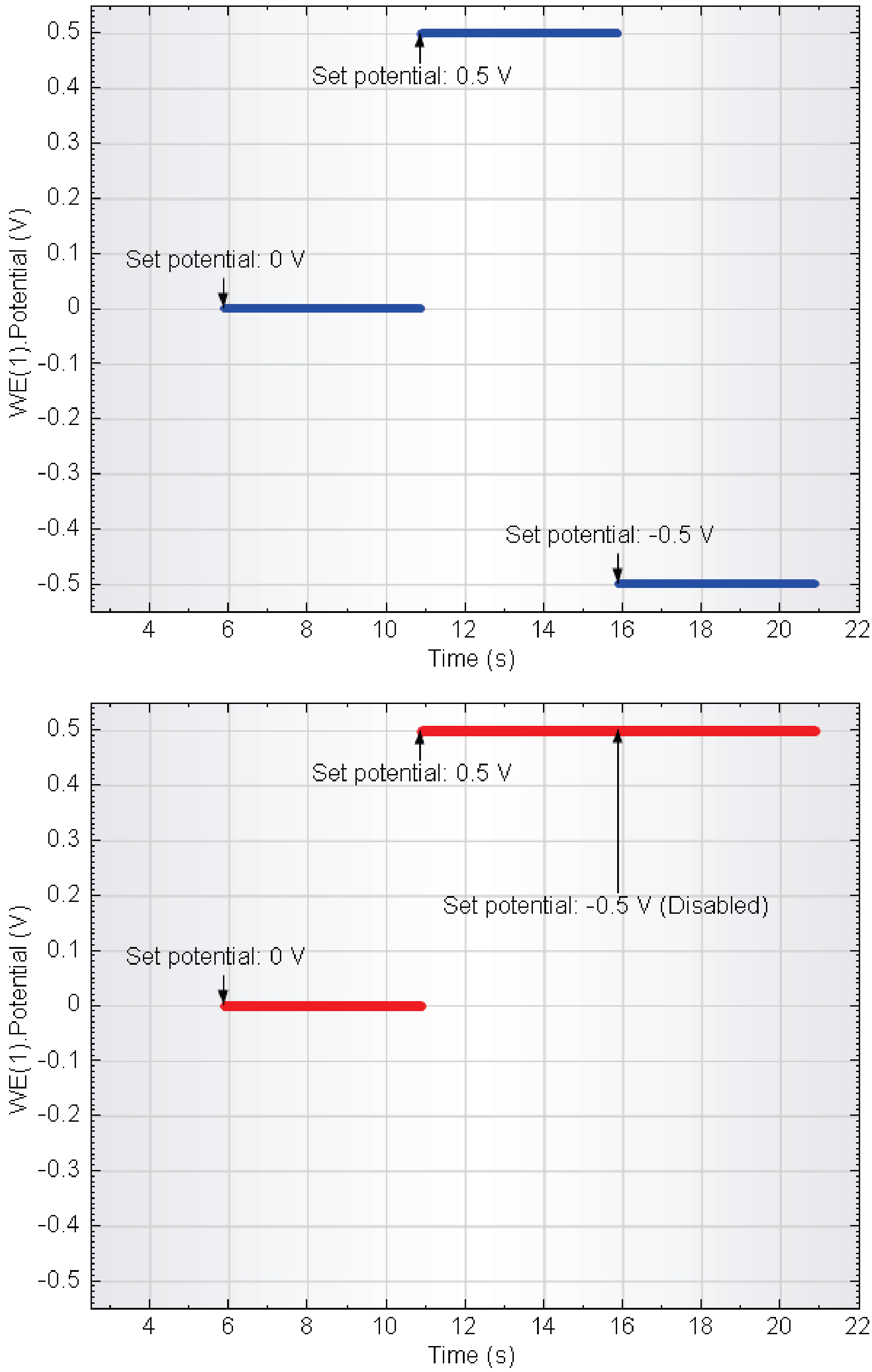


Figure 2.28 – The potential profile used during the measurement on the dummy cell (a) using the original Chrono amperometry procedure (top) and the modified procedure (bottom)

As the third potential step in the procedure has been disabled, NOVA does not execute it, but instead moves to the next command in the procedure, which is a *Record signals (> 1 ms)* command. The response of the cell is measured at a potential of 0.5 V for another five seconds.

If the third *Set potential* command needs to be reactivated, simply right-click the command and set it back to Enabled (see Figure 2.29).

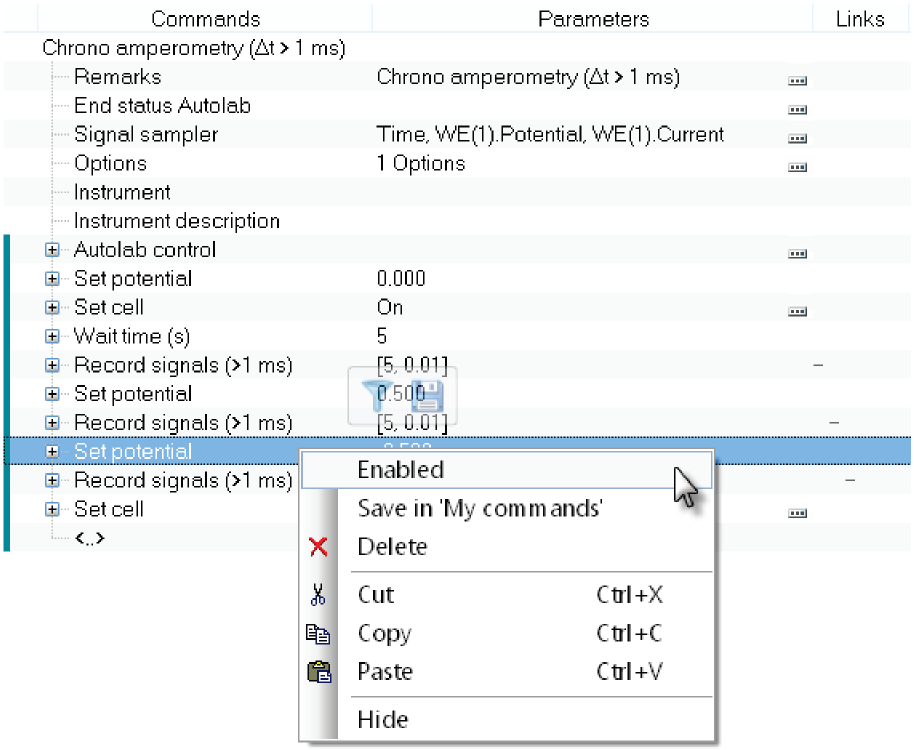


Figure 2.29 – Right-click a disable command to enable it again

2.4.7 – Adding extra commands

The previous examples focused on adding and removing existing commands to or from a procedure. The commands browser, however, displays a large number of commands that can be inserted in a procedure using the same *drag and drop* mechanism described previously. This will be explained in more detail in the next part of this chapter.

This section provides a very simple example for the Autolab cyclic voltammetry potentiostatic procedure.

Reload the procedure into the editor frame and switch the browser frame to commands browser.

It is common practice to purge the solutions with nitrogen before each measurement, in order to get rid of dissolved electroactive gases. Sometimes, measurements are started while the solution is still being purged. An easy way to avoid this is to insert a **reminder** in the procedure. In the commands list, there is a command that does precisely that.

Locate the **Message box** command under the Control list in the browser frame. Drag and drop the *Message box* command at the very beginning of the procedure, before the *Autolab control* command (see Figure 2.30).

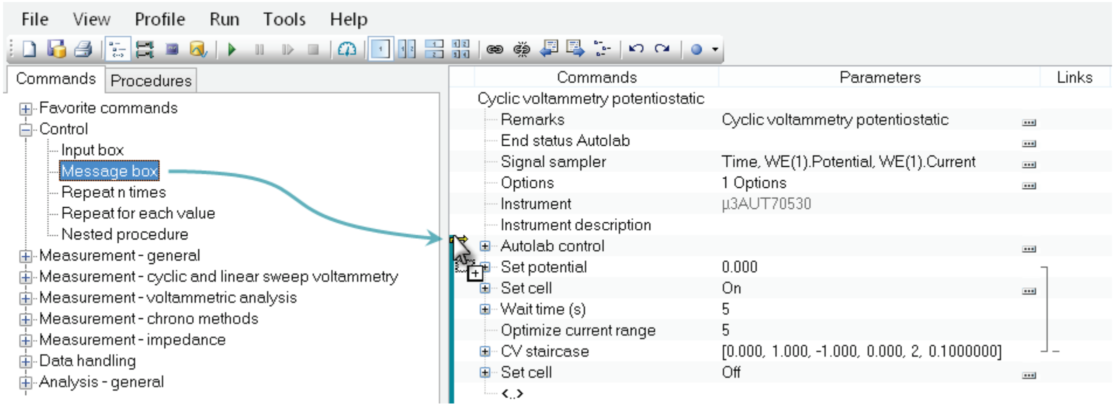


Figure 2.30 – Inserting a Message box to create a reminder

Expand the inserted command. A *Message box* has the following parameters:

- **Title of box:** this is the title of the *Message box*. By default, the Title of box is automatically linked to the Serial number of the instrument (Instrument parameter). This link can be broken if necessary[20]. If no instrument is connected, the Title of box will be unlinked.
- **Message:** this is the message to display in the *Message box*.
- **Time limit (s):** this defines an optional count down for the *Message box*.
- **Use time limit:** defines whether the time limit should be used. When this parameter is set to No, the *Message box* will be displayed indefinitely.

Type in the following strings for the Message parameter (see Figure 2.31):

Message: Switch off the nitrogen purge!

[20] More information on links is provided in Section 2.4.8.

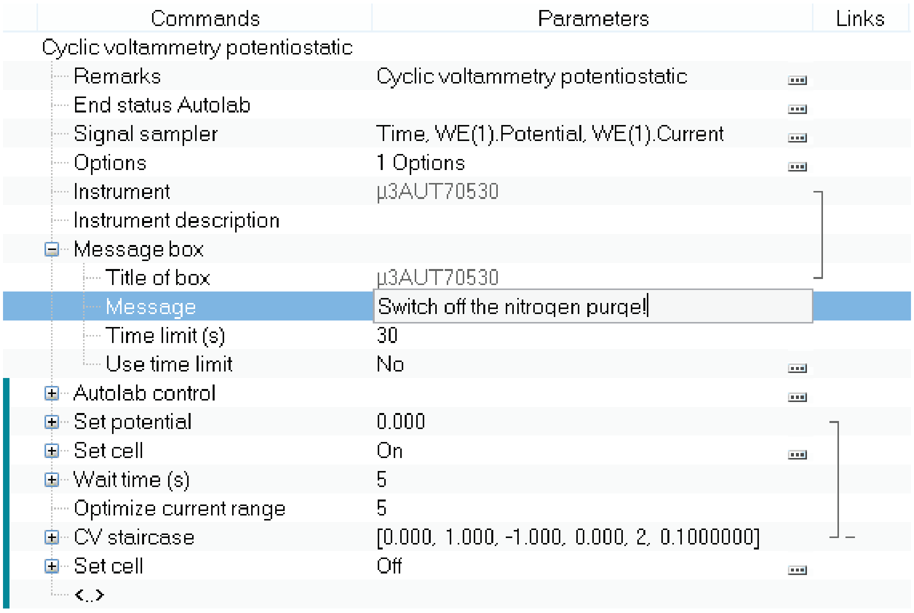


Figure 2.31 – Editing the reminder message

When the measurement start, before the cell is switched on, NOVA will display a window displaying the reminder message (see Figure 2.32).

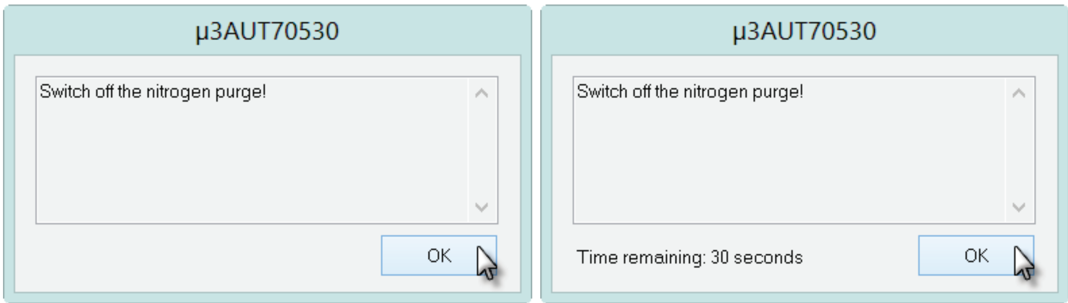


Figure 2.32 – The reminder generated by the *Message box* command (left, Use time limit: off; right, Use time limit: on)

Note

If the Use time limit parameter is set to No, the procedure will not continue until the OK button is clicked. The *Message box* command can therefore be used as an **interrupt** command. It holds the procedure until the user clicks the OK button. *Messages box* commands can be placed anywhere in the procedure.

Note

As explained in Section 2.4.5, the *Message box* command will be added to the Favorite commands group when dragged into the procedure (see Figure 2.33).

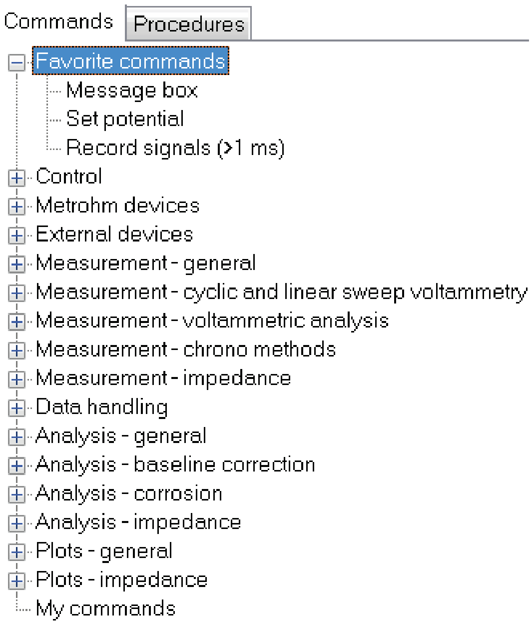


Figure 2.33 – The *Message box* command is added to the Favorite commands group when dragged into the procedure editor

2.4.8 – Linking commands

2.4.8.1 – Understanding links

Linking commands is a very important part of procedure editing in NOVA. A link can be defined as a relationship between two or more parameters in a procedure.

Reload the Autolab Cyclic voltammetry potentiostatic procedure. In the toolbar, click the *Show all links* button to expand the procedure and reveal the existing links (see Figure 2.34). The Autolab Cyclic voltammetry potentiostatic procedure has a total of three links.

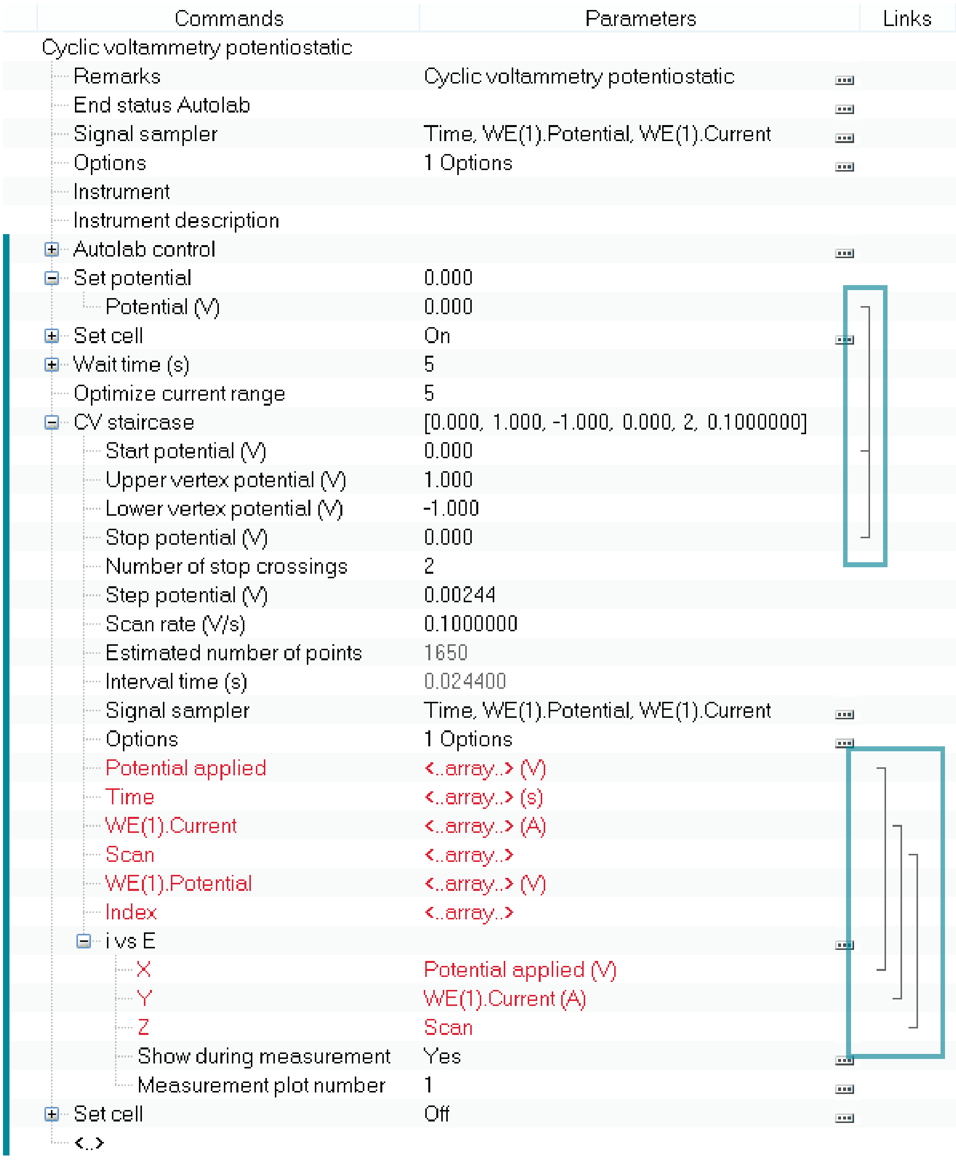


Figure 2.34 – The links used in the Cyclic voltammetry potentiostatic procedure

Links are shown as grey lines on the right-hand side of the procedure editor frame, under the Links column. In the example shown in Figure 2.34, a link is used between Potential parameter of the initial *Set potential* command and the Start and Stop potential parameters of the *CV staircase* command.

Two extra links are used to link the WE(1).Current and the Potential applied signals to the Y and X axis of the **i vs E** plot, respectively. The Z axis is linked to the Index signal.

The three potential values (Set potential, Start potential and Stop potential) are **linked**. Editing one of them will immediately change all of them to the new value. Click the Stop potential value and change it to 0.2 V. Press the enter key to confirm the change. This will not only change the value of the Stop potential, but also of the Start potential and the Set potential value (see Figure 2.35).

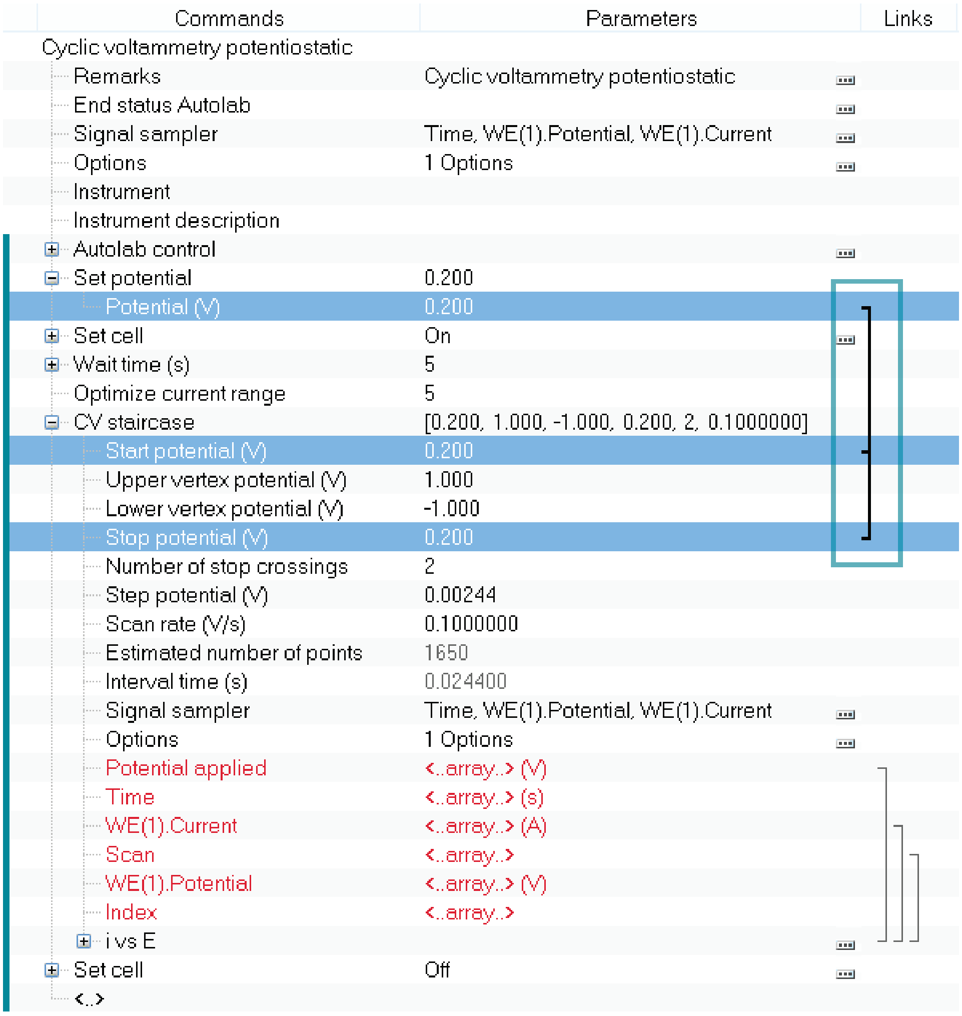


Figure 2.35 – Changing the value of one parameter changes the values of all the other parameters it is linked to

Note

The link connecting the three potential parameters changes from grey to black. This happens whenever one or more parameters or signals that are linked are selected, making it easy to identify the relationship in the procedure.

Existing links can be removed or modified and new links can be created.

2.4.8.2 – Removing links

In the previous example, all three potentials are linked. If the Stop potential should differ from the Start potential and the Set potential value, then the link to the other parameters has to be removed. Click the Stop potential parameter to select it and click the *Unlink selected parameter* button in the toolbar to remove the link (see Figure 2.36).

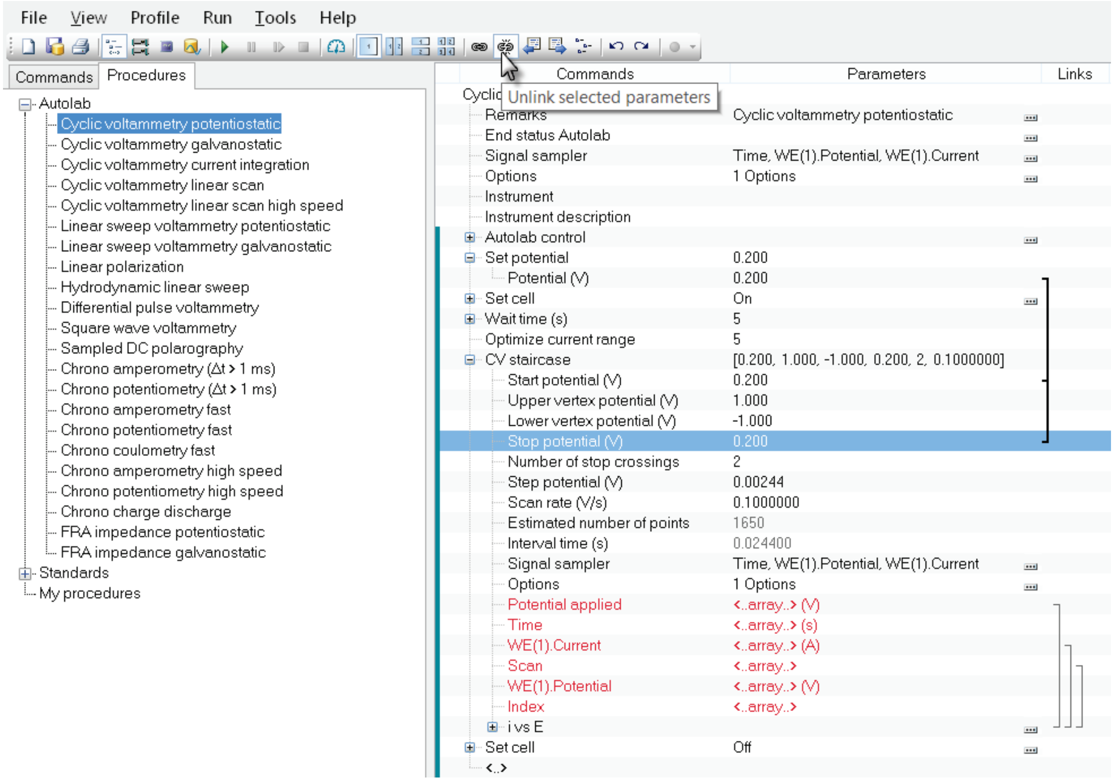


Figure 2.36 – Unlinking the Stop potential

If the Start potential is now set back to 0 V, only the value of the Set potential will be set to the same value. The Stop potential value will remain equal to 0.2 V since it is no longer linked to the two other parameters (see Figure 2.37).

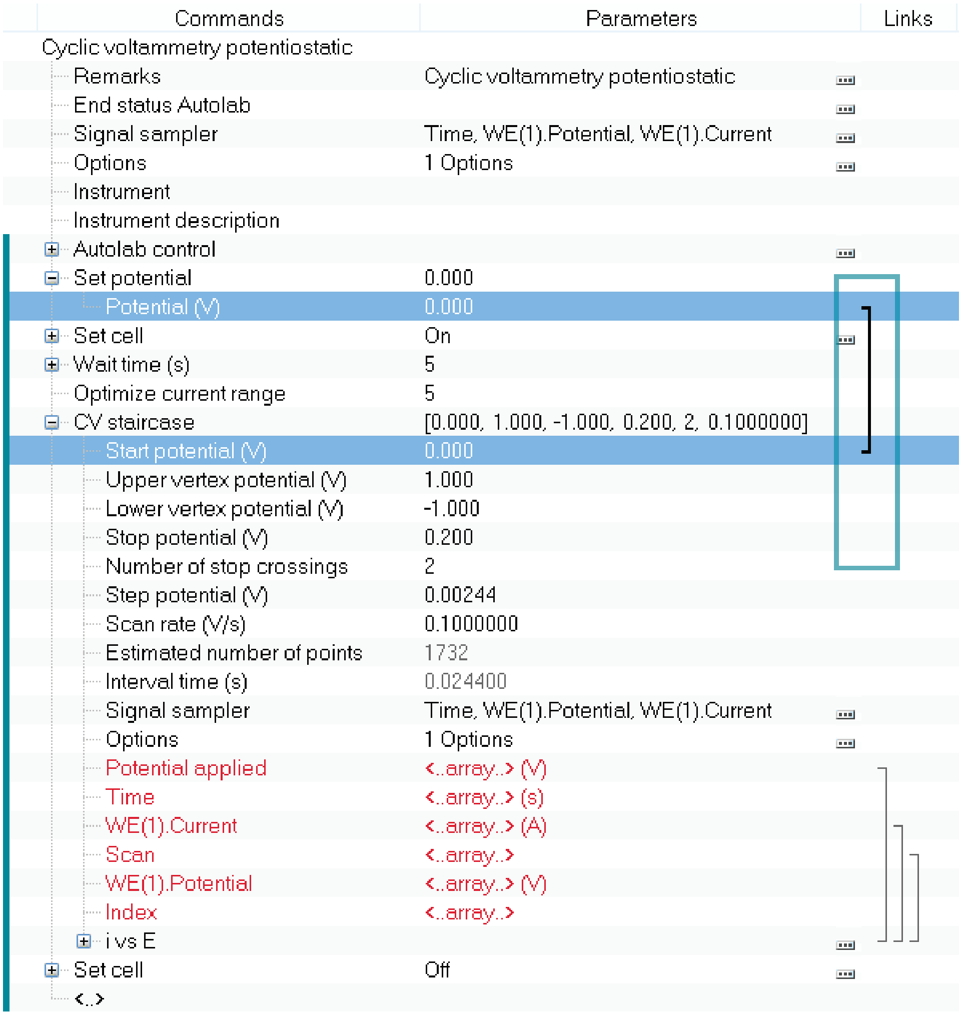


Figure 2.37 – The Stop potential value is now independent

Note

Only one parameter can be unlinked at a time.

Links can also be broken using the right-click menu, or using the CTRL-U keyboard shortcut (see Figure 2.38).

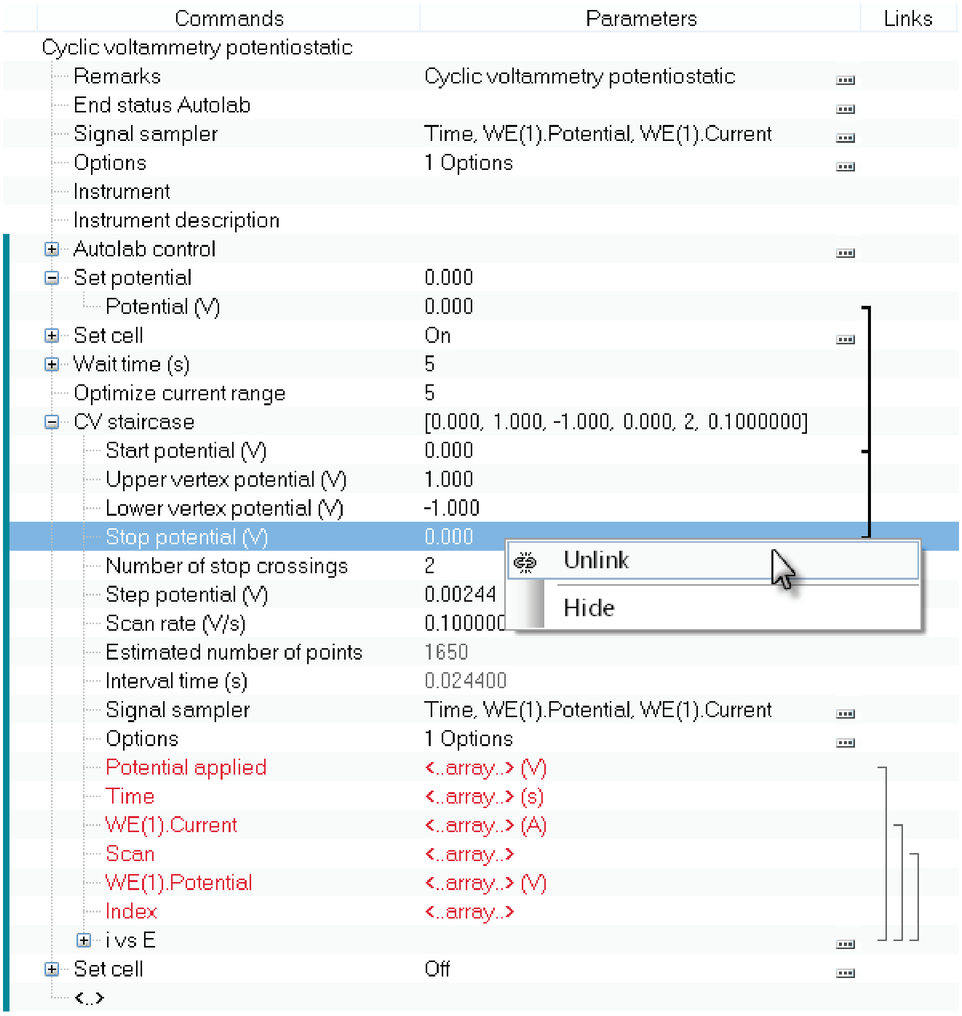


Figure 2.38 – Links can also be broken using the right-click menu or using the CTRL-U keyboard shortcut

2.4.8.3 – Creating links

To create a link, at least **two parameters** must be selected. To select multiple parameters, press and hold the CTRL key on the keyboard and click the parameters that have to be linked in the procedure editor frame.

Note

Links between parameters are not only working during the procedure setup but also during the measurement. This means that, using the links, it is possible to setup dynamic procedures, in which some parameters depend on experimental conditions encountered during the measurements.

Restoring the link between the Stop-, Start- and Set potential values can be achieved by selecting the stop potential and either the start potential, or the value of the set potential or both. Clicking the *Link selected parameters* button in the toolbar will restore the link (see Figure 2.39).

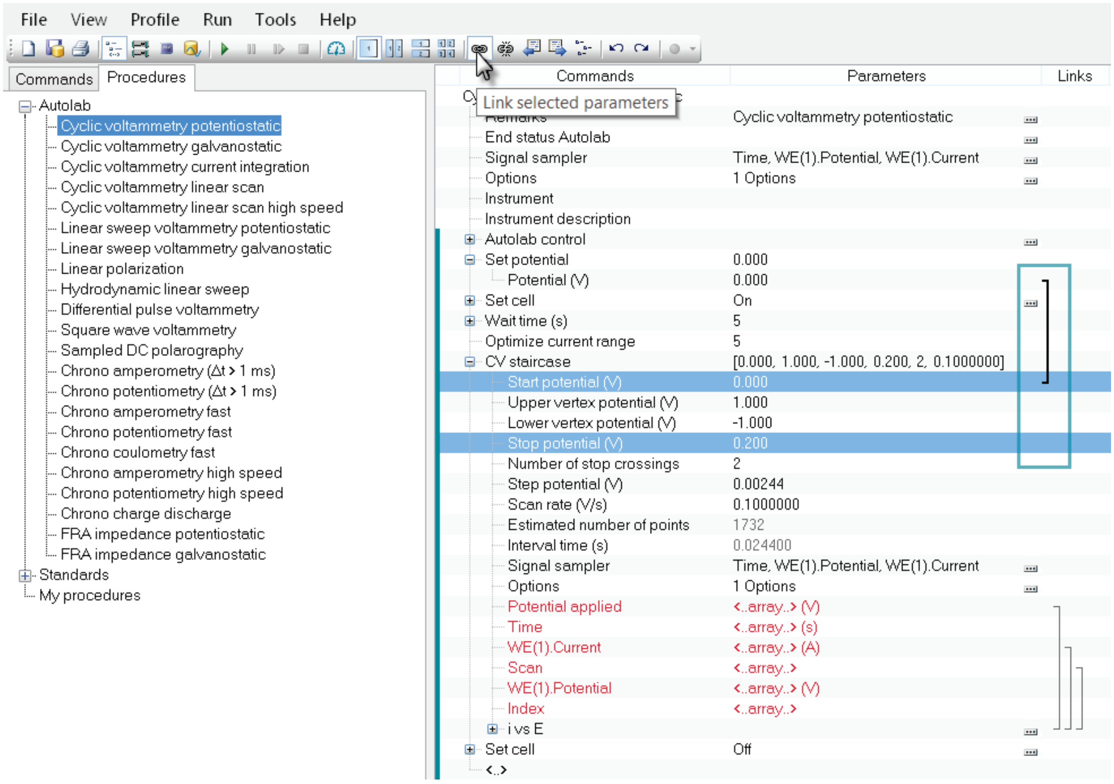


Figure 2.39 – Restoring the link to the stop potential

Note

Removing an existing link requires only one selected parameter. Creating a link, on the other hand, requires at least two selected parameters.

Links can also be created using the right-click menu, or using the CTRL-L keyboard shortcut (see Figure 2.40).

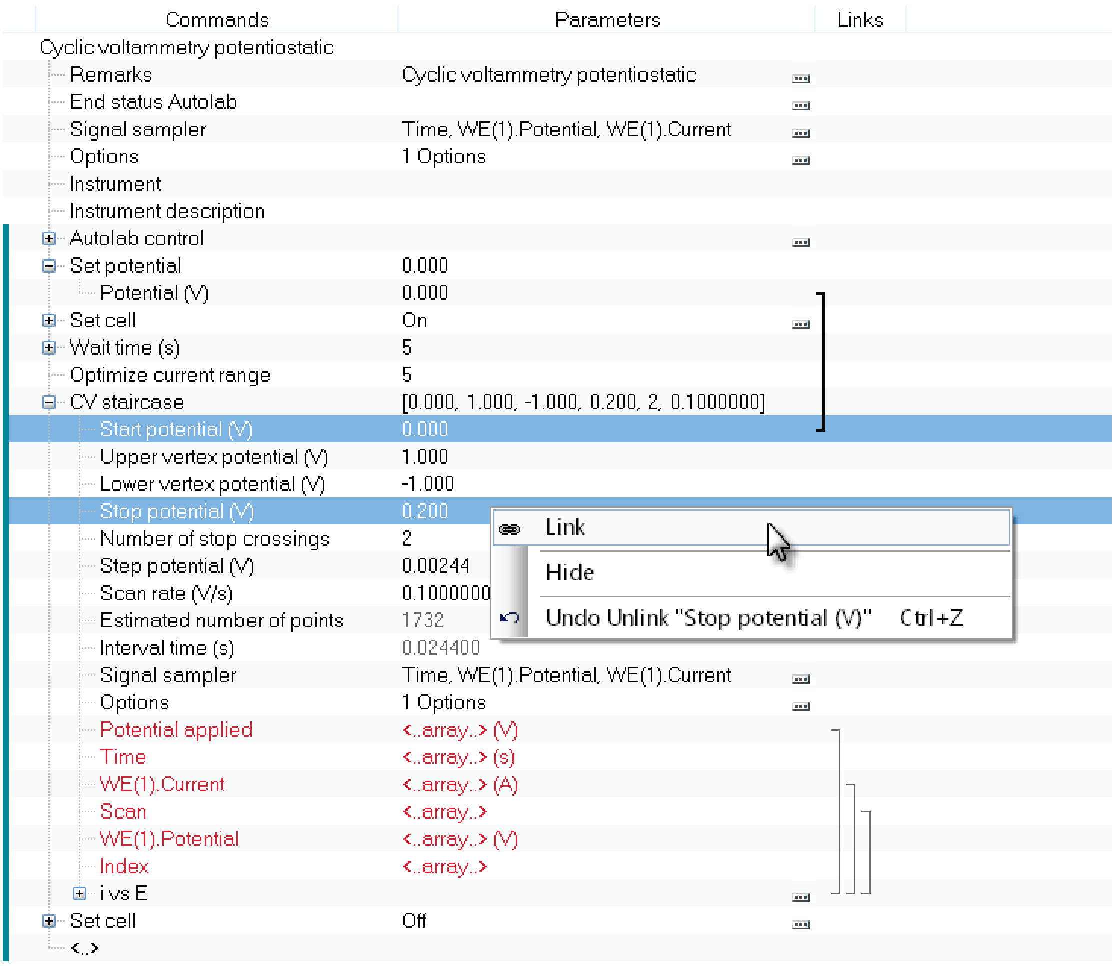


Figure 2.40 – It is possible to create links and remove links using the right-click menu

Note

The Undo Unlink option is also provided in the right-click menu (see Figure 2.40). The Undo and Redo options can be used to restore broken links or break restored links.

2.4.8.4 – Links: the programming tools of NOVA

Links can be used to program in NOVA. Using links, procedure parameters can be linked with one another. Results of data handling steps can be used for plotting or as new parameters for the rest of the experiments. Using links, it is possible to program just about any measurement sequence.

Links can be used to create interactive or dynamic procedures, which allows a change to a procedure parameter *while the experiment is running* depending on parameter values provided by the user or calculated by the software, during the experiment.

A very convenient command called **Input box** is available in the commands browser, under the Control section. The *Input box* works in the same way as the *Message box*.

Reload the Autolab cyclic voltammetry potentiostatic procedure into the procedure editor and select the Input box command from the command browser. Drag and drop it into the procedure, placing it at the very beginning of the procedure (see Figure 2.41).

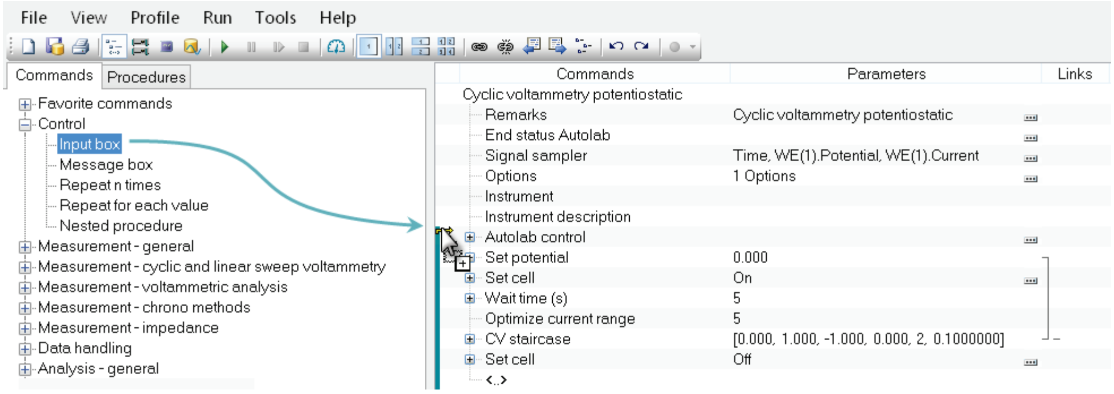


Figure 2.41 – Inserting an Input box in the procedure

Expand the inserted command. An *Input box* has the following parameters:

- **Title of box:** this is the title of the *Input box*. By default, the Title of box is automatically linked to the Serial number of the instrument (Instrument parameter). This link can be broken if necessary[21]. If no instrument is connected, the Title of box will be unlinked.
- **Message:** this is the message to display in the *Input box*.
- **Value:** this is the default value for the input value of the *Input box*.
- **Time limit (s):** this defines an optional count down for the *Input box*.
- **Use time limit:** defines whether the time limit should be used. When this parameter is set to No, the *Input box* will be displayed indefinitely.

In this example, the *Input box* command will prompt the user to enter the requested scan rate for the cyclic voltammogram. The preset value for this procedure is 100 mV/s but this value will be overruled by the user once the procedure is started.

Type in the following parameters (see Figure 2.42):

- **Message:** Enter the value of the scan rate, in V/s
- **Value:** 0.050
- **Time limit (s):** 30
- **Use time limit:** no

[21] More information on links is provided in Section 2.4.8.

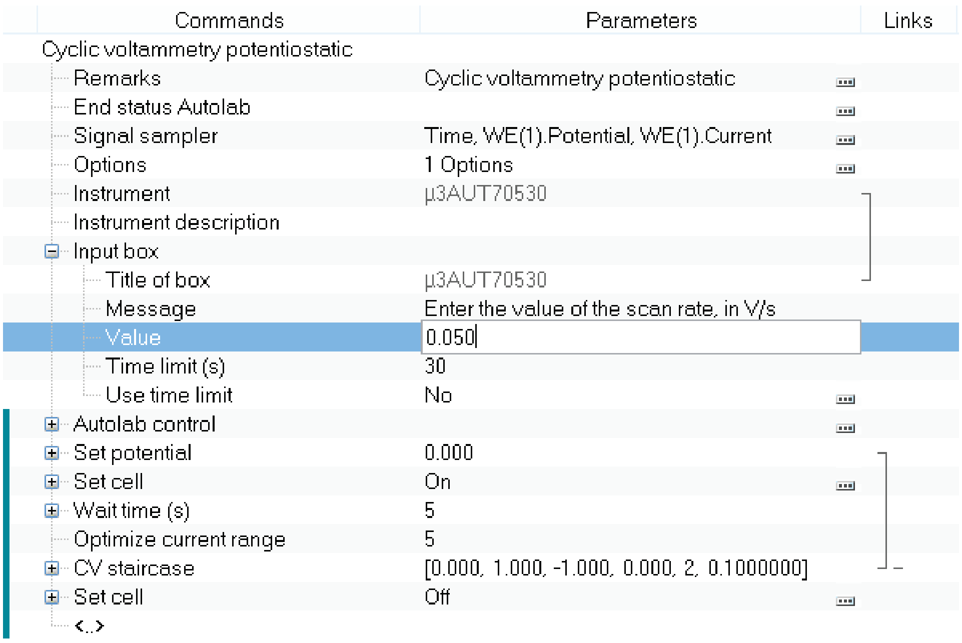


Figure 2.42 – Modifying the Input box parameters

To instruct the software to use the value typed into an Input box, a **link** must be created between the Value parameter of the Input box and the parameter in the procedure it is supposed to replace, in this case, the scan rate.

Creating this link can be done in the same way as in the previous section. Hold the CTRL key, select the Value parameter of the Input box and the Scan rate parameter of the CV Staircase command and press the *Link selected parameters button* in the toolbar (see Figure 2.43).

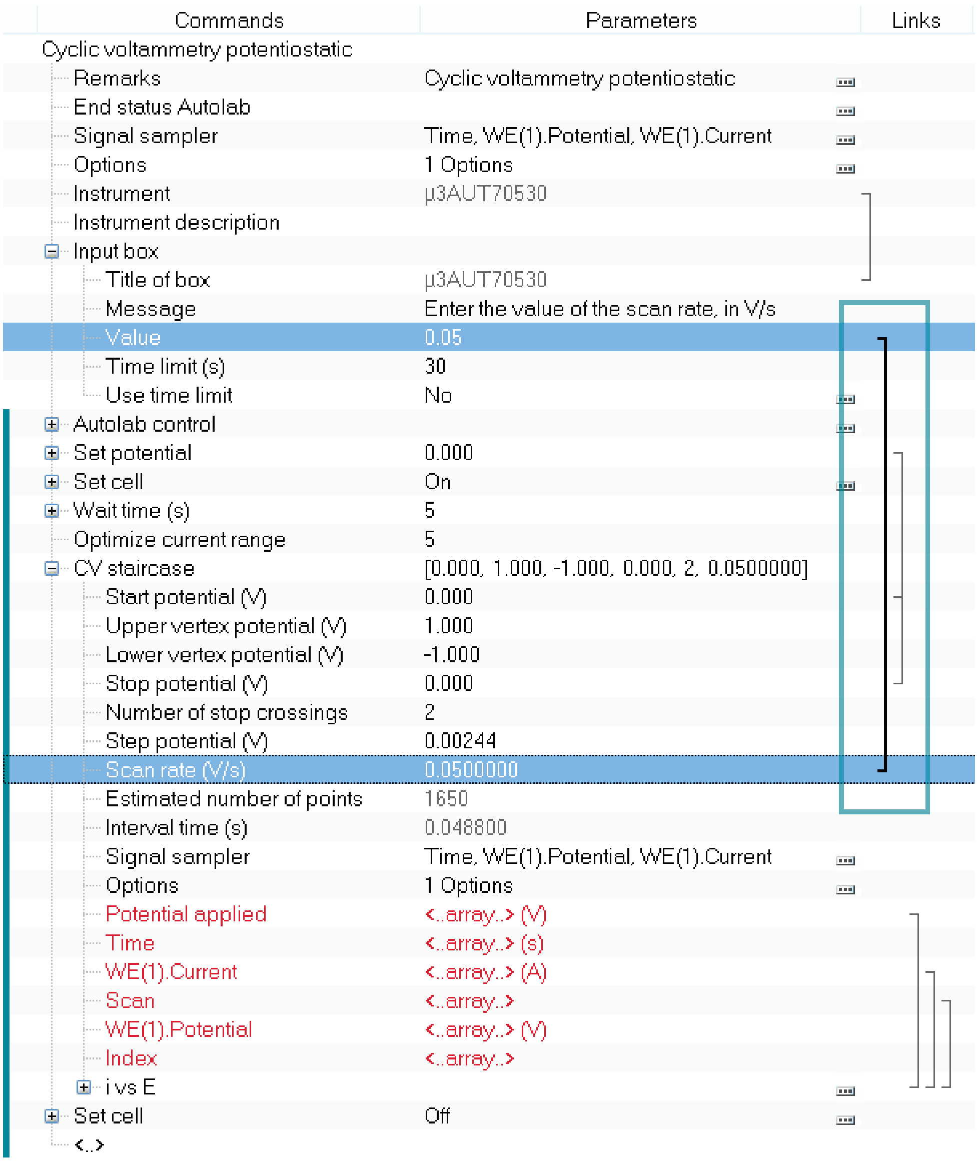


Figure 2.43 – Linking the Input box value to the scan rate

When the link is created, a new black line appears on the right side of the procedure editor frame, linking the value of the Input box to the scan rate. The scan rate also changes from 0.1 V/s to 0.050 V/s when the link is created, thus substituting the preset value with the default value of the *Input box.*

Note

Using *Input box* commands and links allows the creation of interactive procedures. This can simplify research when a procedure has to be repeated a number of times but with different parameters. It also provides a very useful educational tool.

Press the Start button. Instead of starting right away, NOVA will display a window prompting the user to type the required scan rate (Figure 2.44). The default value of 0.05 V/s can be validated by clicking the  button. If another value should be used, a new value can be typed in the Input box.

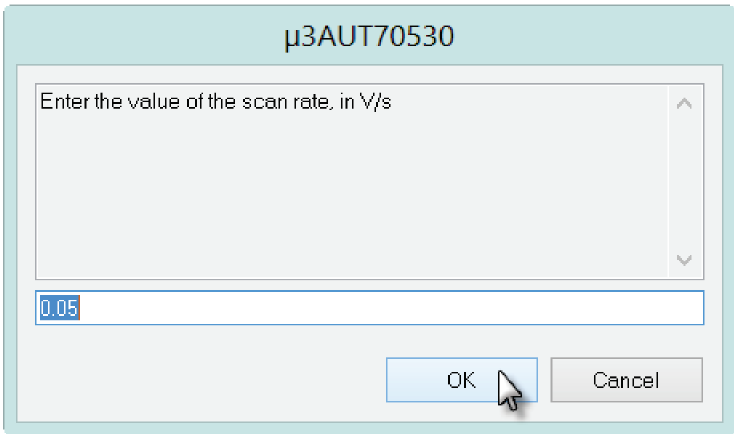


Figure 2.44 – The scan rate value Input box window

2.5 – Advanced procedure editing

This section provides details on advanced editing tools. More attention is given to Timed commands, procedure structure and links.

2.5.1 – Advanced procedures structure

Load the Autolab **Chrono charge discharge** procedure into the editor frame. This is a more complex procedure than the procedures illustrated in the previous sections. The procedure uses a *Repeat n times* command to create a repetition loop. Careful inspection of the procedure reveals that the commands are grouped into three consecutive timed segments, indicated by the green timing guide (see Figure 2.45).

- Segment A is the pre-treatment of the cell.
- Segment B is the bulk of the Chrono charge discharge procedure.
- Segment C is the post-treatment of the cell.

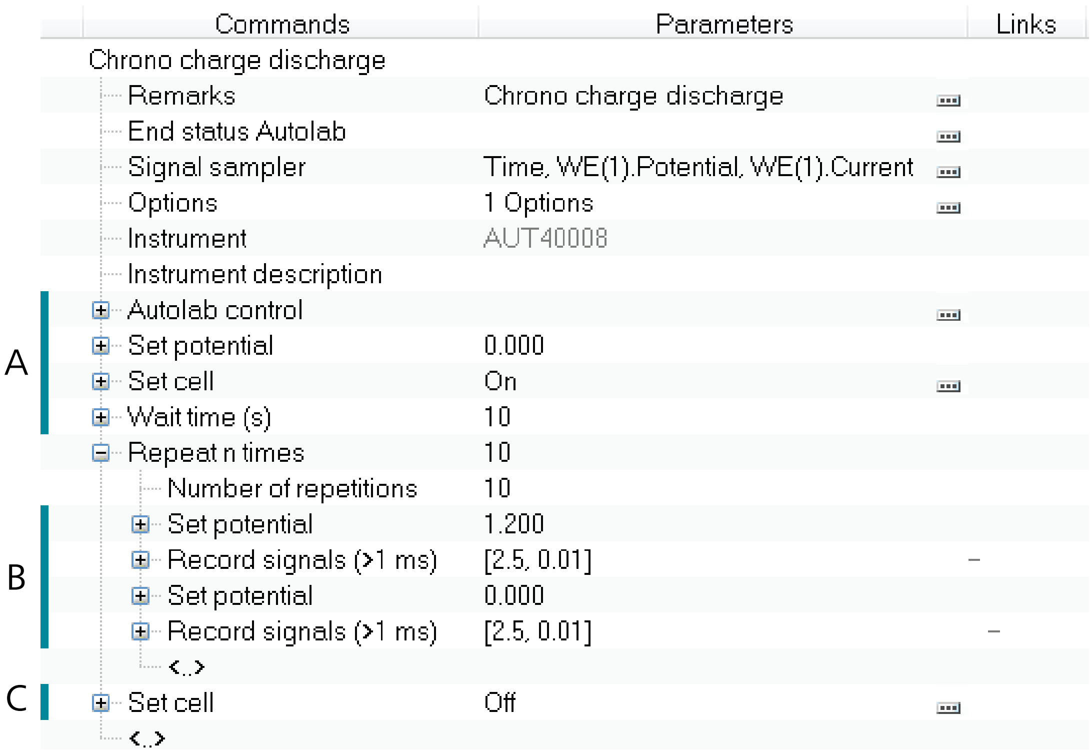


Figure 2.45 – The chrono charge discharge procedure

The first segment (A), the pretreatment, consists of four separate commands:

- Autolab control
- Set potential (0 V)
- Set cell (On)
- Wait time (10 s)

All these commands are timed commands and they will be executed without interruption. To provide a visual indication, a green line is shown on the left-hand side of the procedure editor (see Figure 2.46).

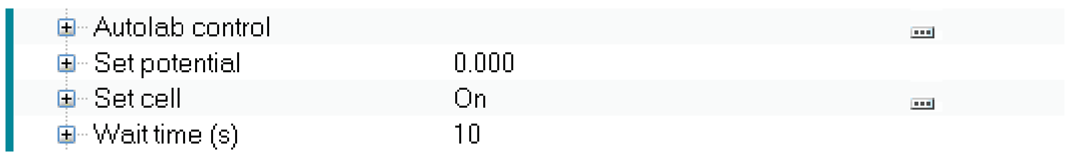


Figure 2.46 – The four commands in section A are all timed commands

The second section (B), which is the measurement itself, contains two different levels (see Figure 2.47).

- The first level is a *Repeat n times* command
- The second level is a series of measurement commands

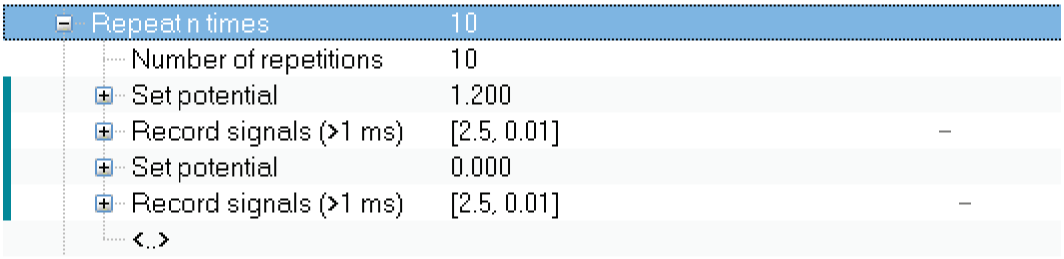


Figure 2.47 – The second part of the procedure contains the repeat loop and the measurement sequence

The *Repeat n times* command creates a repeat loop. Since this command is not a timed command, it creates a break in the green timing guide, indicating that there will be an interruption at each repetition.

Commands that are placed inside the *Repeat n times* command will be repeated *n* times when the procedure is executed. However, this is only true for those commands that are located on the second level under the *Repeat n times* command.

All the commands that are located under the *Repeat n times* command will be repeated.

The four commands located inside the *Repeat n times* command are all timed commands and they will be executed without interruption. This is again indicated by the green timing guide located on the left-hand side of the procedure editor (see Figure 2.47).

During the measurement, the two potential steps (*Set potential* command) and the two chrono measurements (*Record signals (>1 ms)* command) will be executed without timing interference from background Windows activity.

The final segment of this procedure consists of a single command that turns the cell off when the measurement is finished (see Figure 2.48). Since this is again a timed command, the timing guide will be shown on the left hand side.

Figure 2.48 – The final segment of the procedure is used to switch the cell off at the end of the measurement

The timing of the full procedure is interrupted at two locations, as indicated by the timing guide on the left-hand side of the procedure editor (see Figure 2.49).

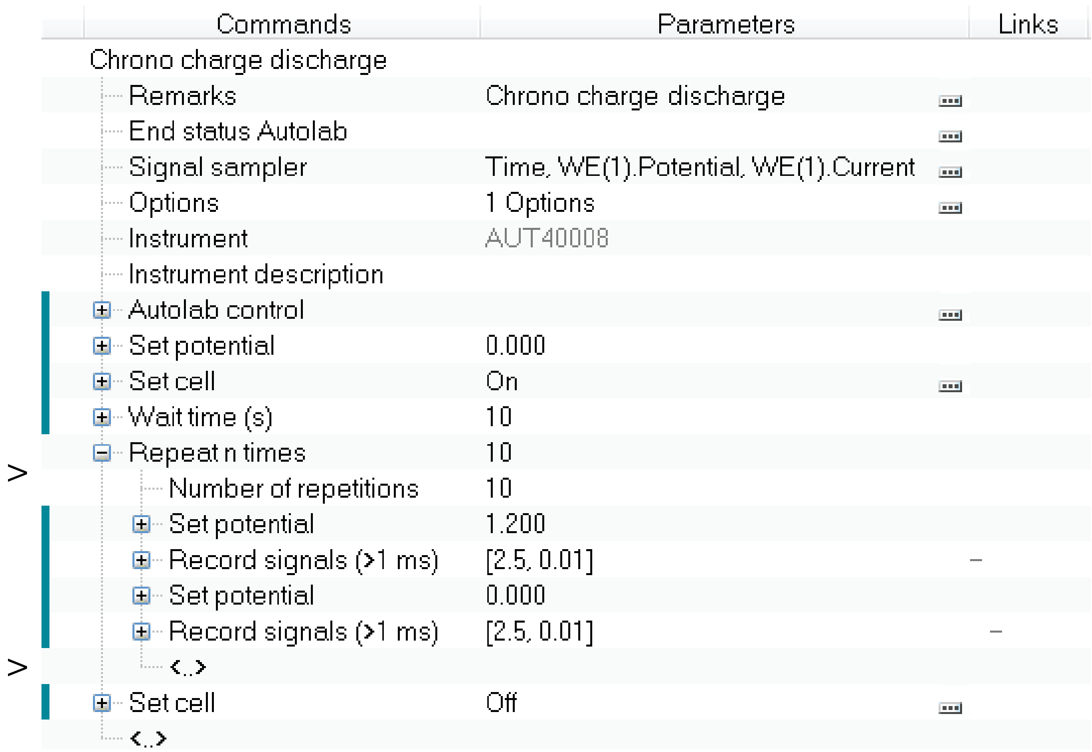


Figure 2.49 – The timing of the procedure is interrupted at the beginning and at the end of the *Repeat n times* command

The interruptions are located before and after the *Repeat n times* command. Since the *Repeat n times* command is not a timed command, it creates a small interruption in the measurement timing. This interruption will be observed at each repetition. Depending on the activity of the host computer, the interruption can be in the range of a few seconds.

Figure 2.50 shows the potential profile used in the standard chrono charge discharge procedure. A sequence of steps is repeated ten times during the measurement. The potential changes from 0 V to 1.2 V.

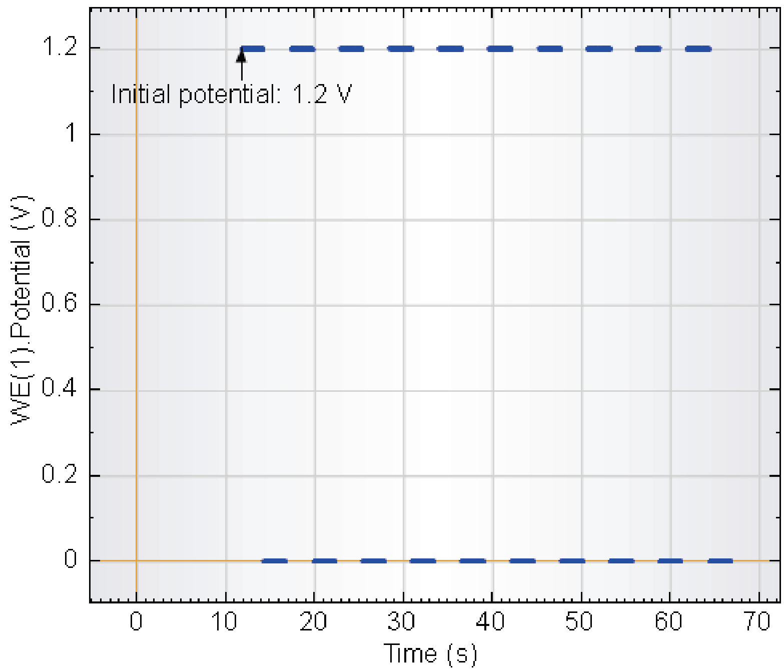


Figure 2.50 – The potential profile used in the standard chrono charge discharge procedure

Detailed inspection of the data shown in Figure 2.50 shows that the time delay between two recorded steps within the same repeat loop is 0.012 s (30.979 s – 30.967 s) whereas the time delay between two consecutive steps located in two different repetitions is ~ 0.6 s (17.385 s – 16.773). This illustrates the timing difference and the time delay introduced by untimed commands like a *Repeat n times* command (see Figure 2.51).

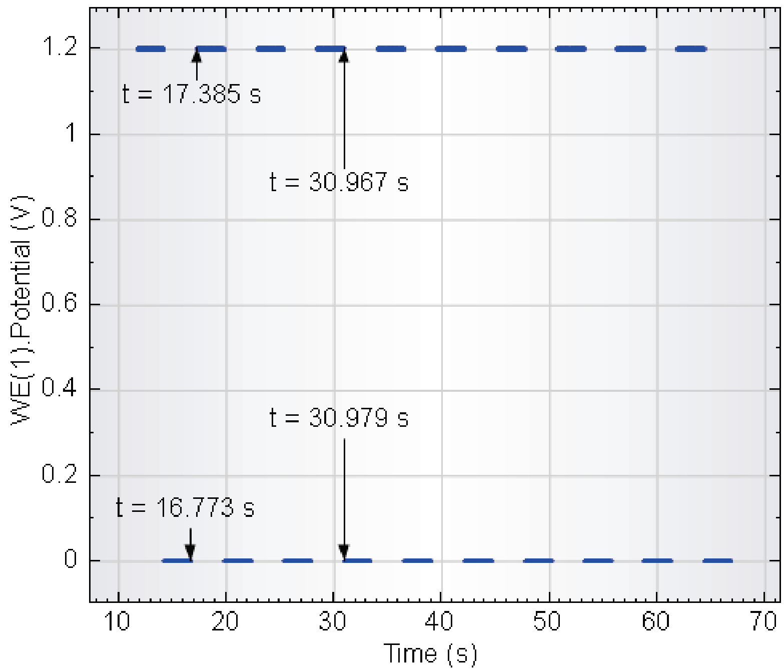


Figure 2.51 – Illustration of the timing difference in NOVA

Note

The time between timed commands will always be very accurately determined by the internal clock of the Autolab. The time delay introduced by untimed commands will be affected by the background activity of the host computer and will therefore be less predictable.

2.5.2 – Building advanced procedures

The final section of this chapter describes how to use the procedure editor to create an advanced procedure from scratch.

Click the button to clear the procedure editor frame. The editor will display an almost empty procedure editor frame (see Figure 2.52).

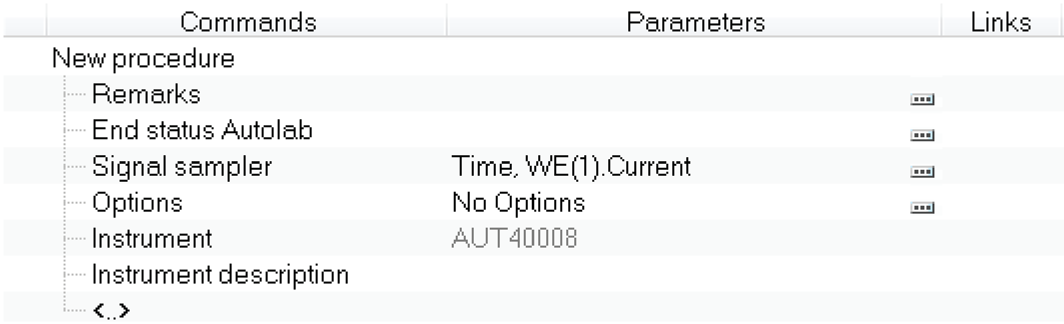


Figure 2.52 – The empty procedure editor frame

The seven lines displayed in the procedure editor frame are the default components of each procedure.

> **Note**
>
> The default Signal sampler includes the Time and WE(1).Current (optimized) signals. The default Options are set to No Options. Please refer to Sections 1.3.3 and 1.3.4 for more information on the Signal Sampler and the Options, respectively.

In this section, the step-by-step construction of an advanced procedure will be illustrated. The intention is to create a procedure that performs a Linear sweep voltammetry measurement at four different, pre-defined, scan rates. This procedure will be designed with a preconditioning stage.

2.5.3 – Defining the preconditioning stage

The preconditioning of the working electrode for this example involves four consecutive steps:

1. Setting the instrument to potentiostatic mode and selecting the initial current range[22]
2. Setting the potential to the preconditioning value, 0 V
3. Switching the cell On
4. Waiting for 5 seconds
5. Finding the most suitable current range

The commands required to perform these actions are all located in the **Measurement – general** group of the commands browser:

1. Autolab control
2. Set potential
3. Set cell
4. Wait time
5. Optimize current range

Select the *Autolab control* command and drag it into the procedure editor (see Figure 2.53).

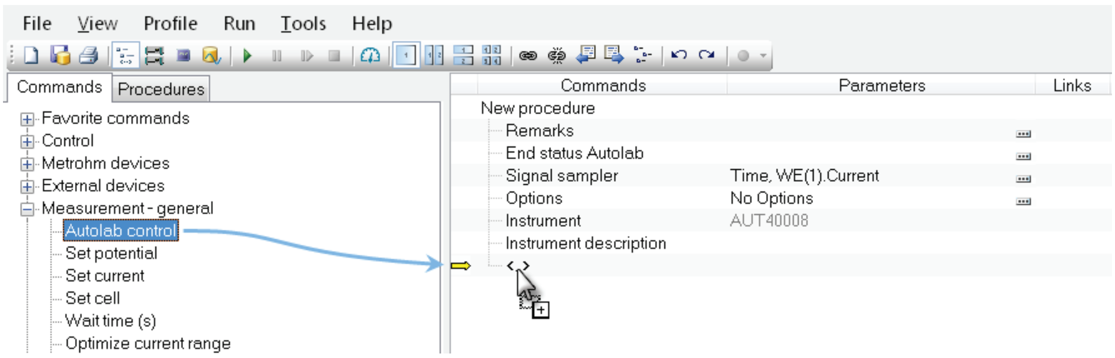


Figure 2.53 – Adding the Autolab control command

To set the parameters of the Autolab control command, click the ⋯ button located on the right-hand side of the procedure editor window (see Figure 2.54).

[22] It is a good habit to set the instrument to the correct settings at the beginning of a procedure.

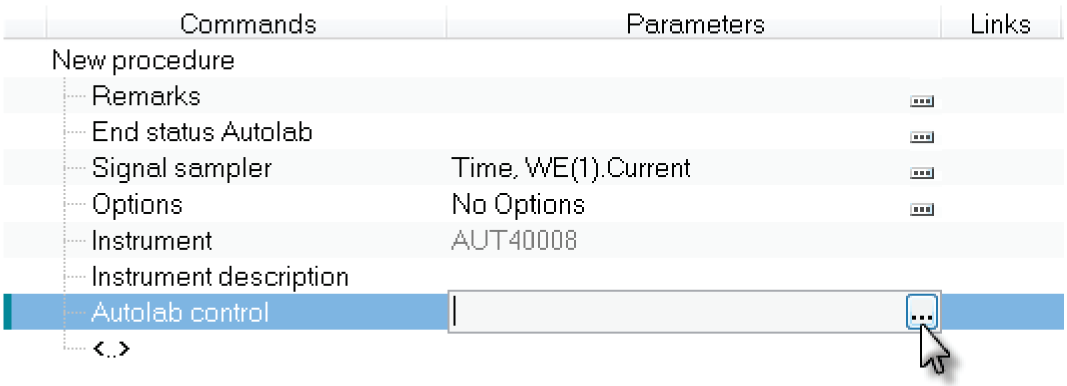


Figure 2.54 – Opening the Autolab control settings

A new window, called Autolab control, will be displayed (see Figure 2.55). This window displays the settings currently defined on the connected instrument and can be used to change any of the instrument settings at any time during a procedure. In Figure 2.55, the basic settings for the main potentiostat/galvanostat can be defined[23].

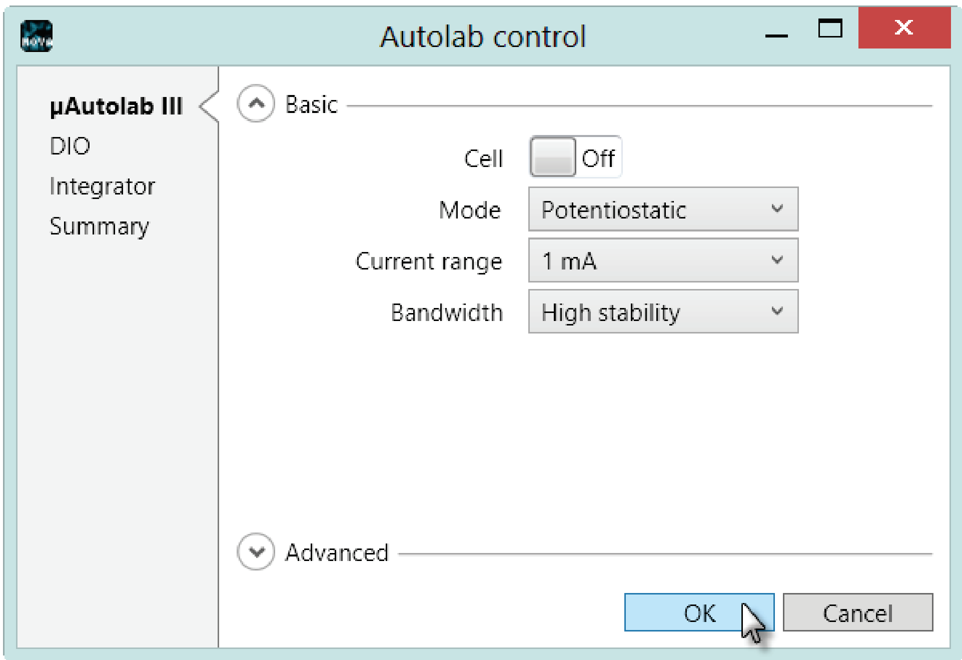


Figure 2.55 – The Autolab control window

For this procedure, the Autolab control command will be used to set the instrument to potentiostatic mode and in the 1 mA current range. Using the drop-down menus, set the Mode and the Current range settings to Potentiostatic and 1 mA, respectively (see Figure 2.56).

[23] The available Autolab settings depend on the hardware configuration.

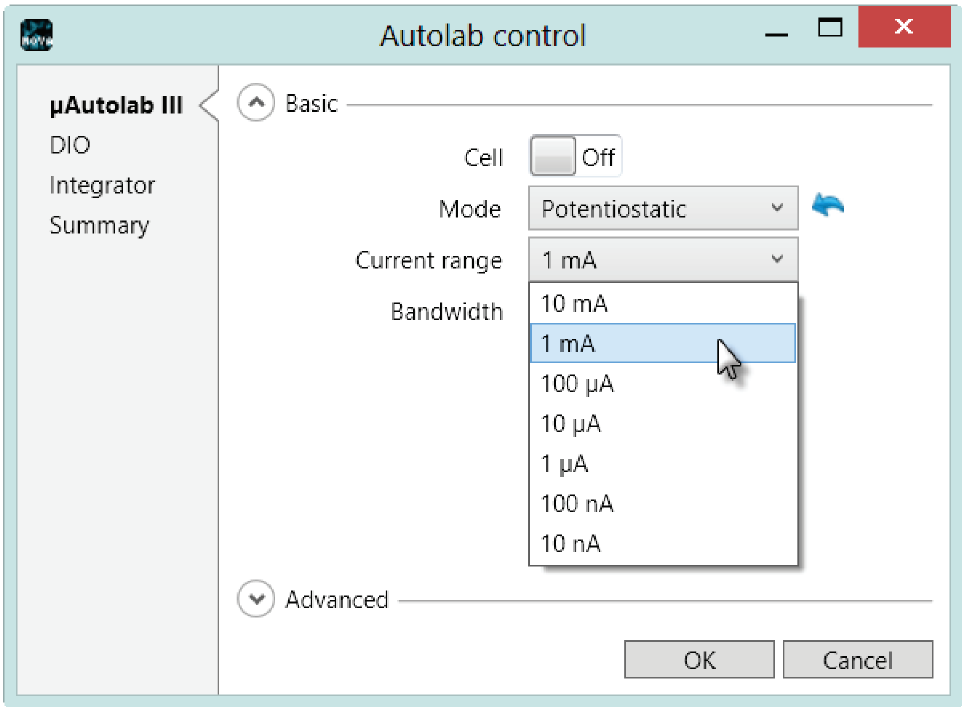


Figure 2.56 – Using the Autolab control command to set the instrument to Potentiostatic mode and in the 1 mA current range

Click the OK button to close the Autolab control window. The settings specified Figure 2.56 will now be displayed in the procedure editor, below the Autolab control command (see Figure 2.57).

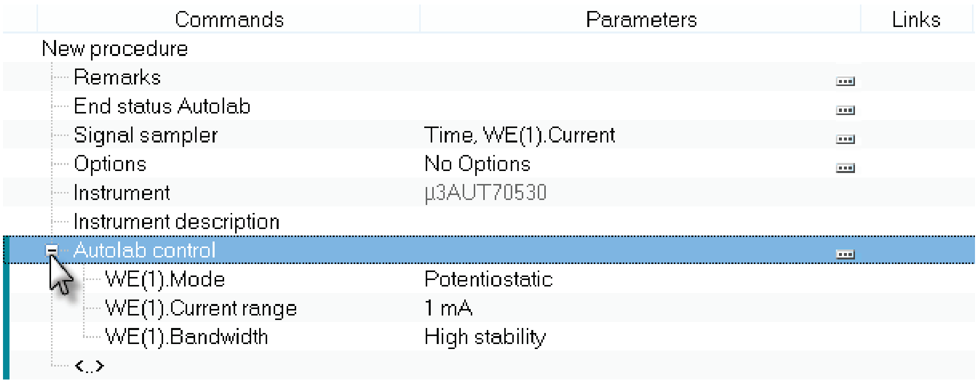


Figure 2.57 – The Autolab control settings are displayed in the procedure editor

Next, locate the *Set potential*, *Set cell*, *Wait time* and *Optimize current range*[24] commands and drag them into the procedure editor, under the *Autolab control* command (see Figure 2.58).

[24] For more information, please refer to the Command list document, available from the Help menu.

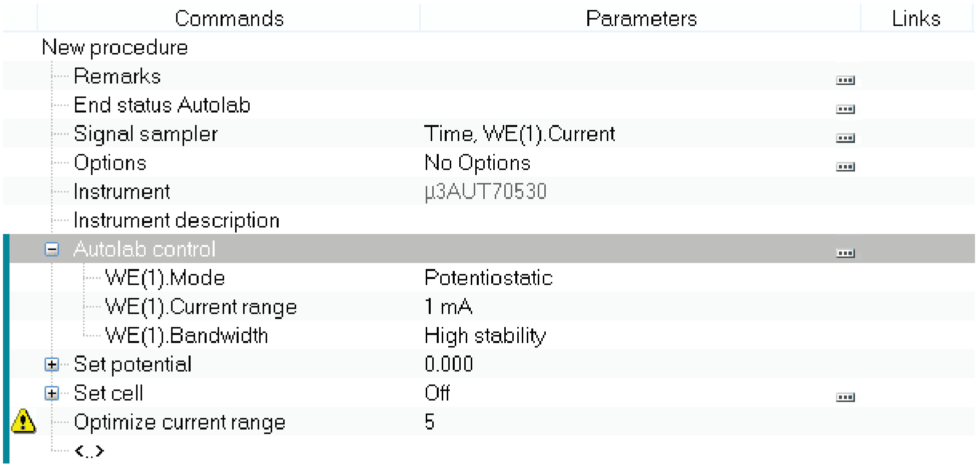


Figure 2.58 – Adding the Set potential, Set Cell, Wait time and Optimize current range commands

A warning symbol (⚠) will be visible in the procedure editor, next to the *Optimize current range* command (see Figure 2.58). This warning symbol only appears after adding the *Optimize current range* command to the sequence.

A toolip is available for each warning in the procedure editor (see Figure 2.59).

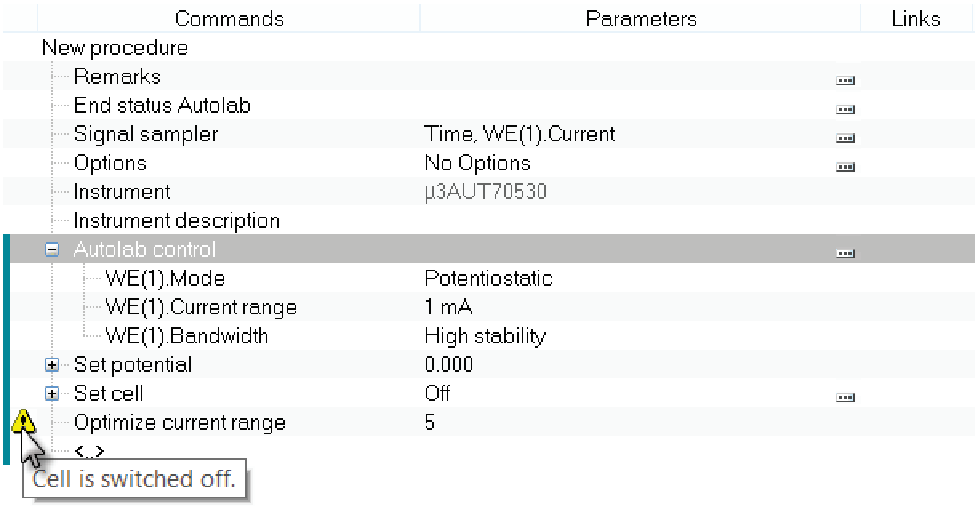


Figure 2.59 – A tooltip is available for each warning in the procedure editor

Note

To provide help in the procedure building process, validation of the procedure is performed in the background, in real time. Whenever a problem is detected, NOVA will display a warning symbol (⚠) next to the command(s) afftected by this problem. Whenever an error is detected, NOVA will display an error symbol (⛔) next to the command(s) affected by this error.

The default parameter of the *Set cell* command is Off. Since this command must be used to switch the cell On, the command has to be expanded and the parameter changed from Off to On (see Figure 2.60).

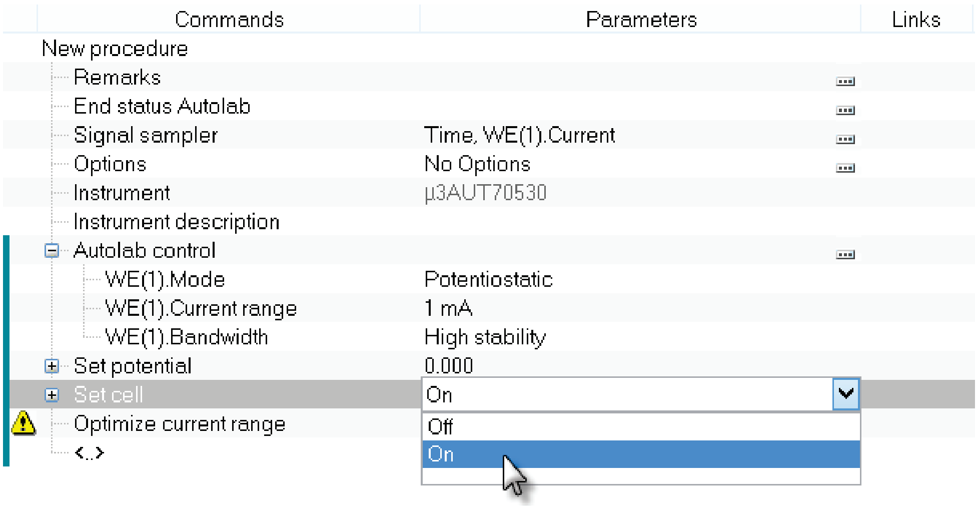


Figure 2.60 – Changing the settings of the Cell status parameter

Note

As soon as the cell is set to On status in the procedure editor, the warning symbol ( ) next to *Optimize current range* command is cleared since the warning is no longer valid (see Figure 2.61).

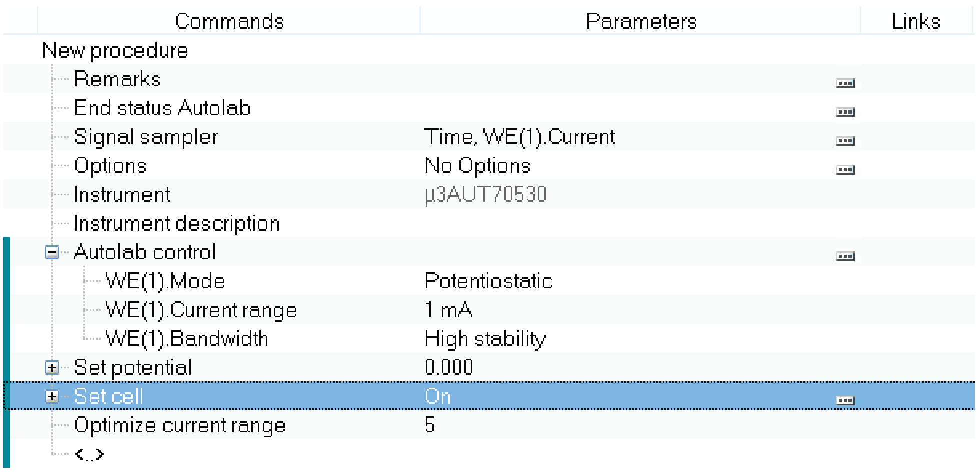


Figure 2.61 – The warning is removed when the Cell status is switched to On in the procedure editor

The value of the preconditioning potential is not yet defined. It will be linked to the start potential of the linear sweep voltammetry command which means that its value is not relevant at this time.

2.5.4 – Defining the measurement conditions

For this example, four consecutive linear sweep voltammetry measurements will be performed, using four pre-defined scan rates.

For this part of the procedure, the following steps are required:

1. Start a linear sweep voltammetry measurement using the first defined scan rate
2. Repeat the previous measurement for the three other scan rates

To perform this measurement, it is possible to use a procedure in which four *LSV staircase* commands are used, one after the other. This is however quite cumbersome, as the parameters of each command would have to be edited individually.

On the other hand, the same results could be obtained by inserting a pre-defined *LSV staircase* command into a repeat loop and by changing the scan rate after each repetition.

The *Repeat for each value* command from the **Control group** has been designed specifically for this task. In this example, it will be used in combination with the *LSV staircase* command.

Select the *Repeat for each value* command from the browser and drag it in the procedure editor frame, under the preconditioning stage (see Figure 2.62).

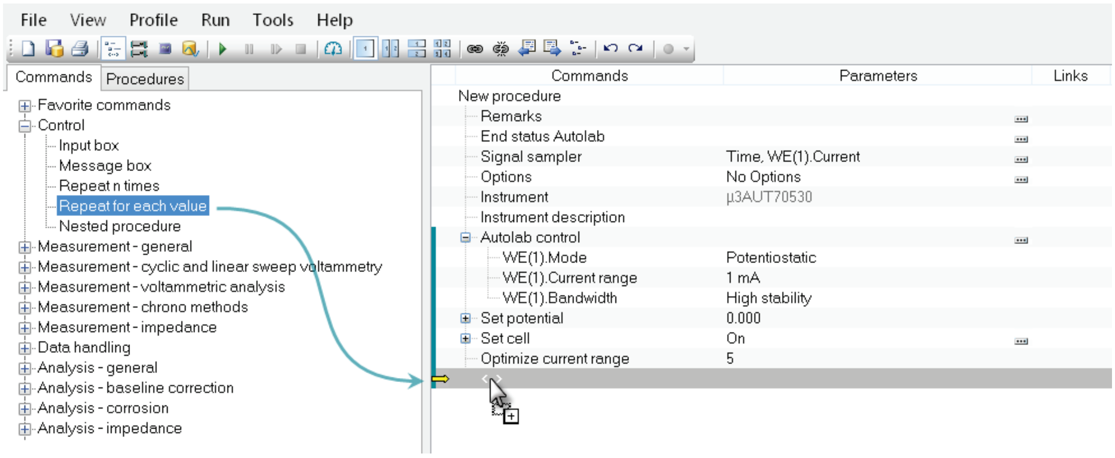


Figure 2.62 – Adding the *Repeat for each value* command to the procedure

The *Repeat for each value* command creates a repeat loop, for which the number of repetitions is equal to the number of pre-defined values of the command.

Add the *LSV staircase* command, from the **Measurement – Cyclic and linear sweep voltammetry** group to the *Repeat for each value* command. Next, add an additional *Set potential* command to the sequence, as shown in Figure 2.63.

Finish the sequence by adding a *Wait time (s)* command, at the end of the repeat loop.

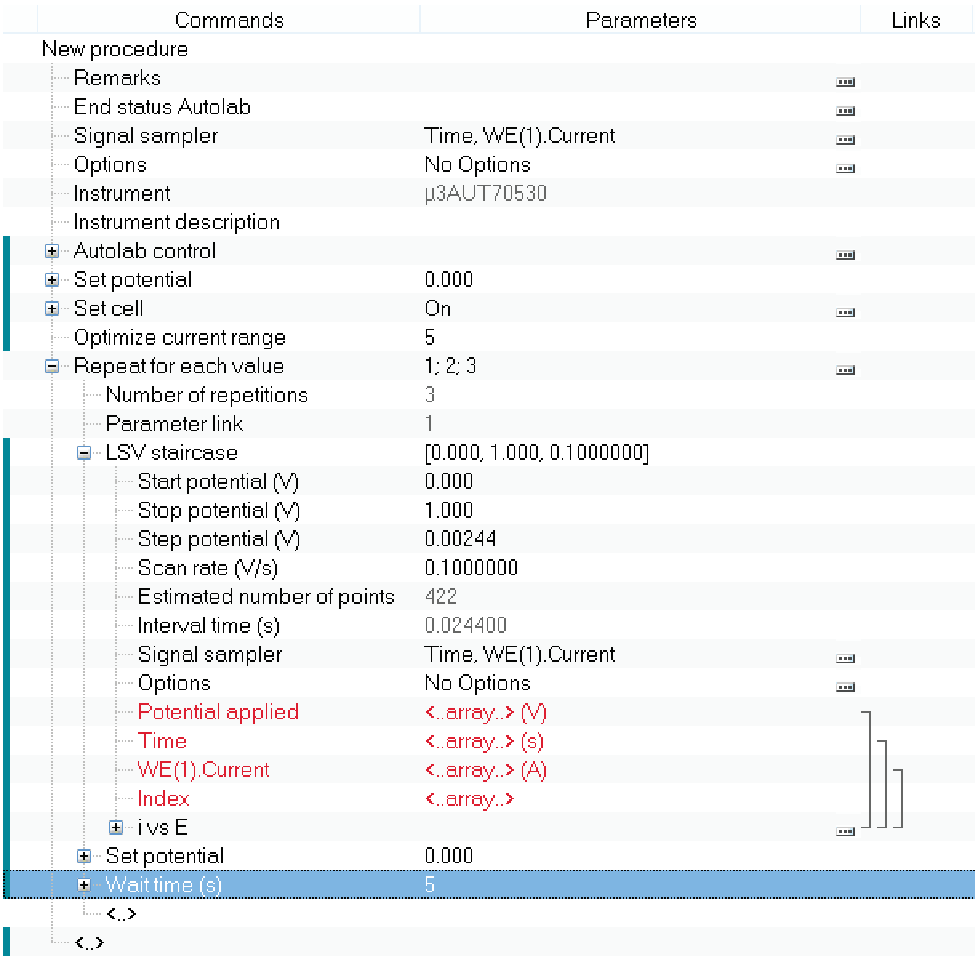


Figure 2.63 – Adding the *LSV staircase*, *Set potential* and *Wait time* commands to the procedure

For this example, the Start potential of the *LSV staircase* command will be -0.06 V and the Stop potential will be set to 0.55 V. The start potential value will also be used as the preconditioning potential.

Create a link between the Potential parameter of the *Set potential* command used in the preconditioning stage and in the repeat loop and the Start potential parameter of the *LSV Staircase* command (see Figure 2.64). Change the values of the Start potential and the Stop potential of the *LSV staircase* command to -0.06 V and 0.55 V, respectively (see Figure 2.64).

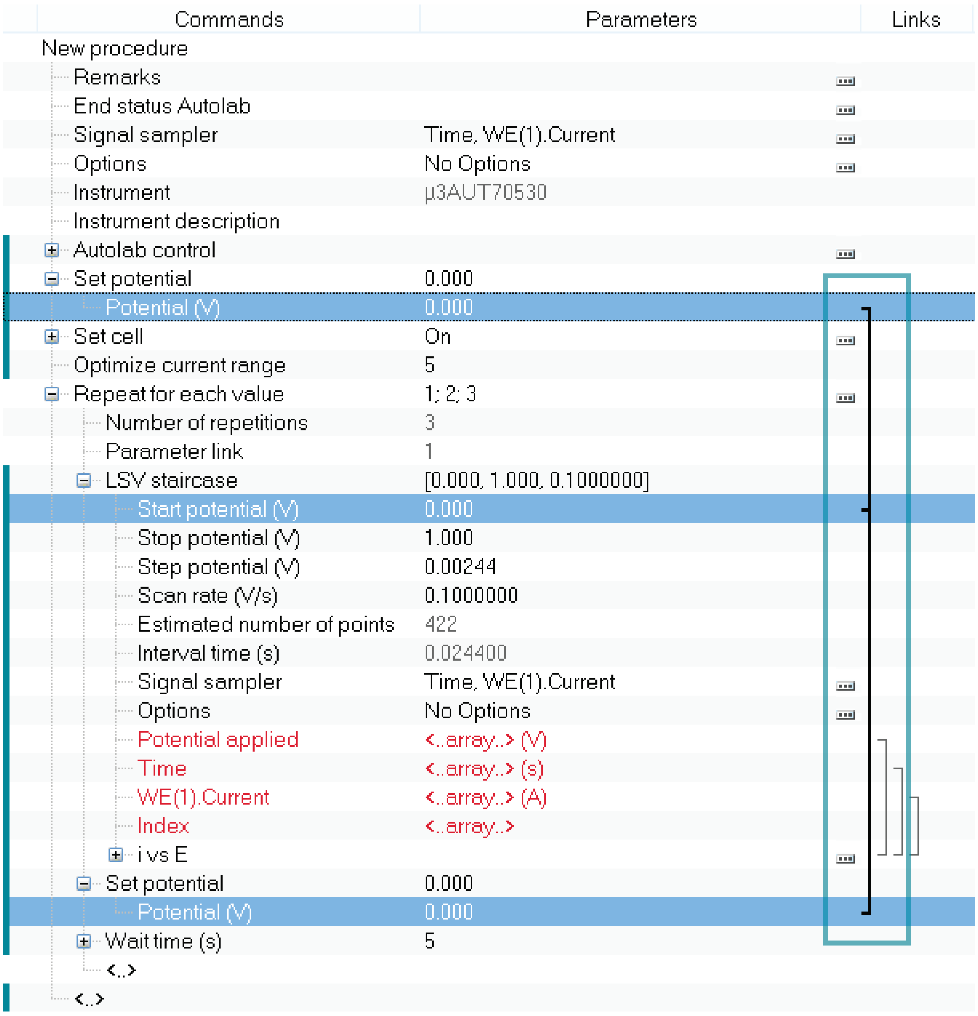


Figure 2.64 – Modifying the *LSV staircase* parameters and creating the link with the preconditioning potential

The *Repeat for each value* command has three default pre-defined values (1, 2 and 3). To change these values, click the … button located on Values parameter line (see Figure 2.65).

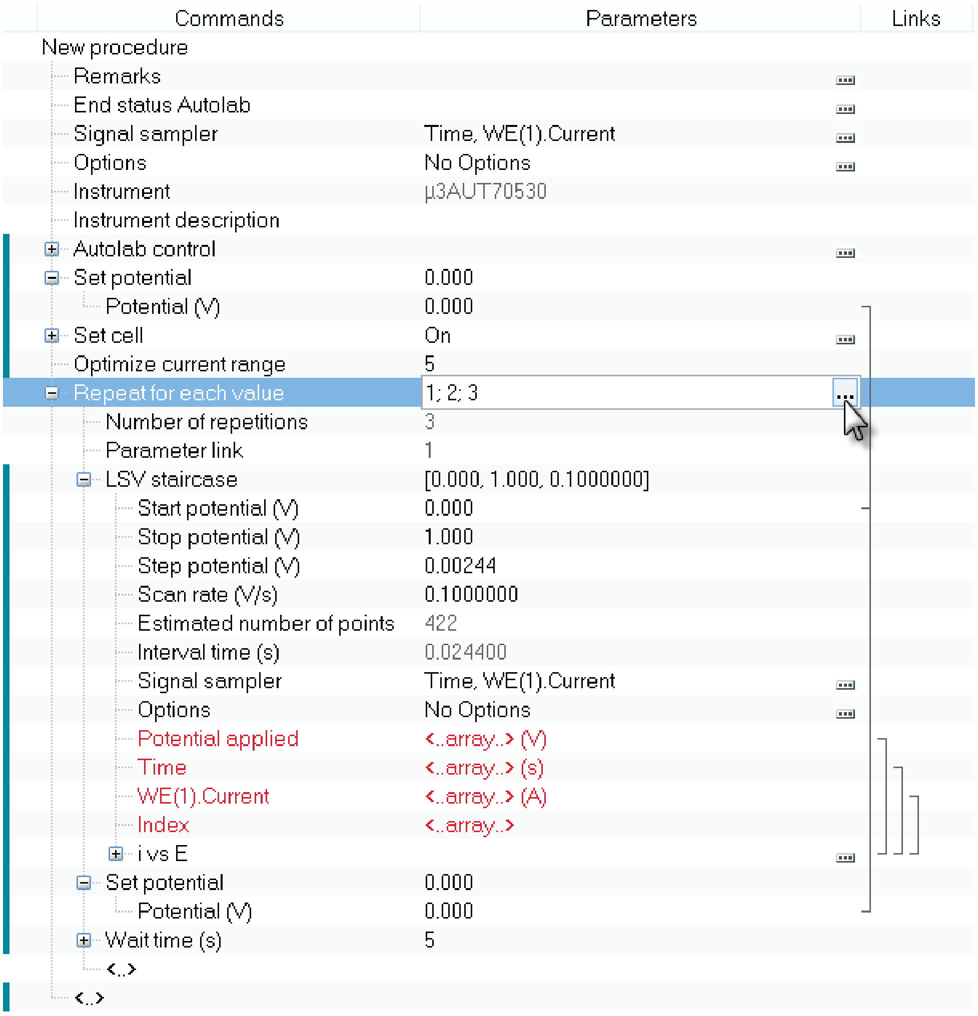


Figure 2.65 – Adjusting the *Repeat for each value* command

This will open a **Range builder** window that can be used to construct a sequence of values to be linked to the scan rate of the *LSV staircase* command (see Figure 2.66).

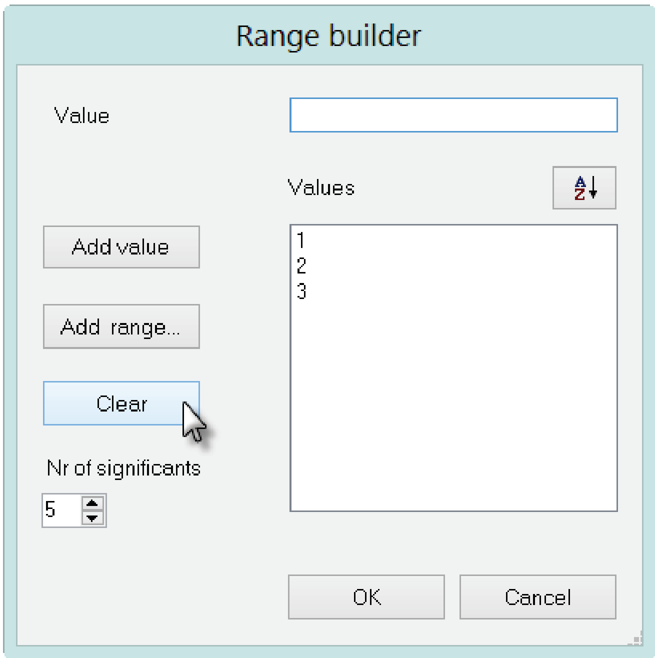


Figure 2.66 – Opening the Range builder window

The Range builder is a useful tool that can be used to edit the sequence of the Repeat for each value command. There are two ways to add values to the range.

- The first option is to type each individual value in the Value field on the top of the window and click the Add value button.
- The second option is to use the Range generator, which is selected by pressing the Add range... button. This option is suitable for adding a large number of values to the range.

Note

The Range builder provides a Clear button to remove any previously added values and a sort button which can be used to sort the values of the range.

Click the Clear button to remove the default values from the range and click the Add range... button to display the Range generator window (see Figure 2.67).

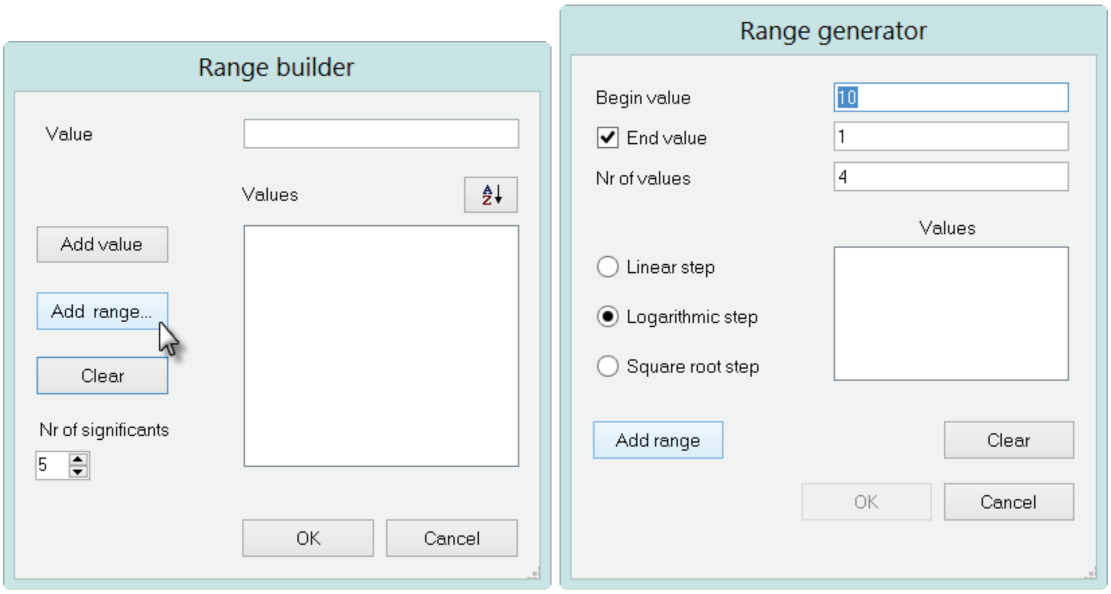


Figure 2.67 – Pressing the Add range... button displays the Range generator window

The Range generator is used to create a list of values using the following parameters:

- **Begin value:** the first value of the list
- **End value:** the final value of the list
- **Nr of values:** the total number of values in the list
- **Step type:** the distribution of the values in the list (linear, logarithmic or square root)

> **Note**
>
> If a list of identical values needs to be created, the End value checkbox can be unchecked. When this is done, only the Begin value will be used to create the list.

For this example, we are going to use a total of four values, ranging from 0.050 V/s to 0.5 V/s, with a square root distribution. Type these parameters in the range generator window and press the Add range button to generate the list of values, click the OK button to close the generator window and return to the Range builder window. The generated list of values will be displayed in the Values field of the Range builder window (see Figure 2.68).

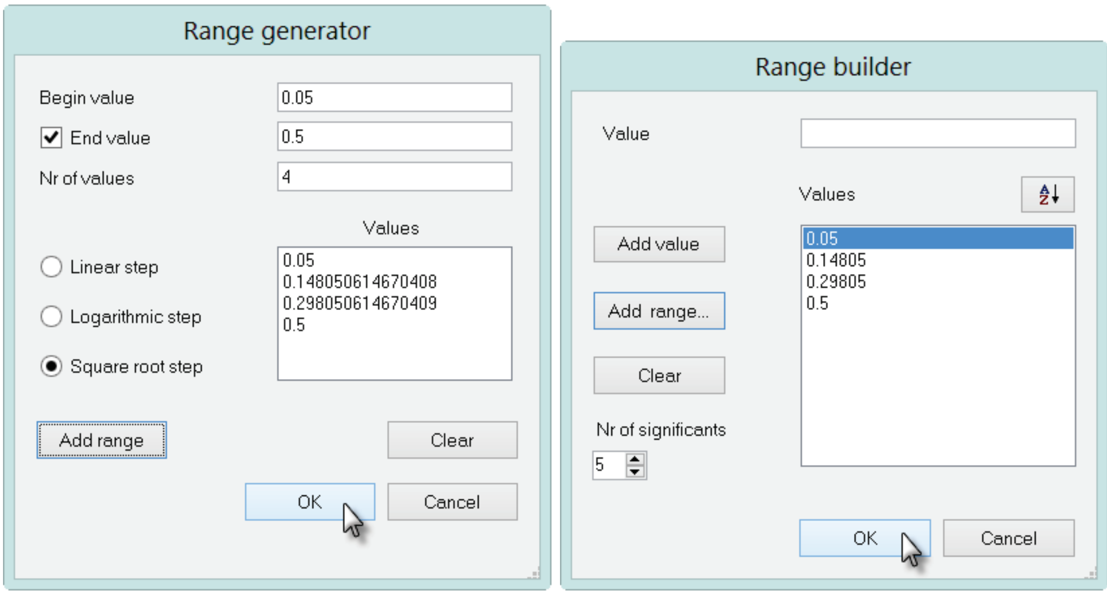


Figure 2.68 – Closing the Range generator window will update the contents of the Range builder window

Note

It is possible to sort the list in ascending or descending order by clicking the button located in the Range builder window.

Click the OK button to return to the procedure editor. The *Repeat for each value* command will be updated (see Figure 2.69). To complete the setup of the repeat loop, the values of the command have to be linked to the Scan rate parameter of the *LSV staircase* command (see Figure 2.69).

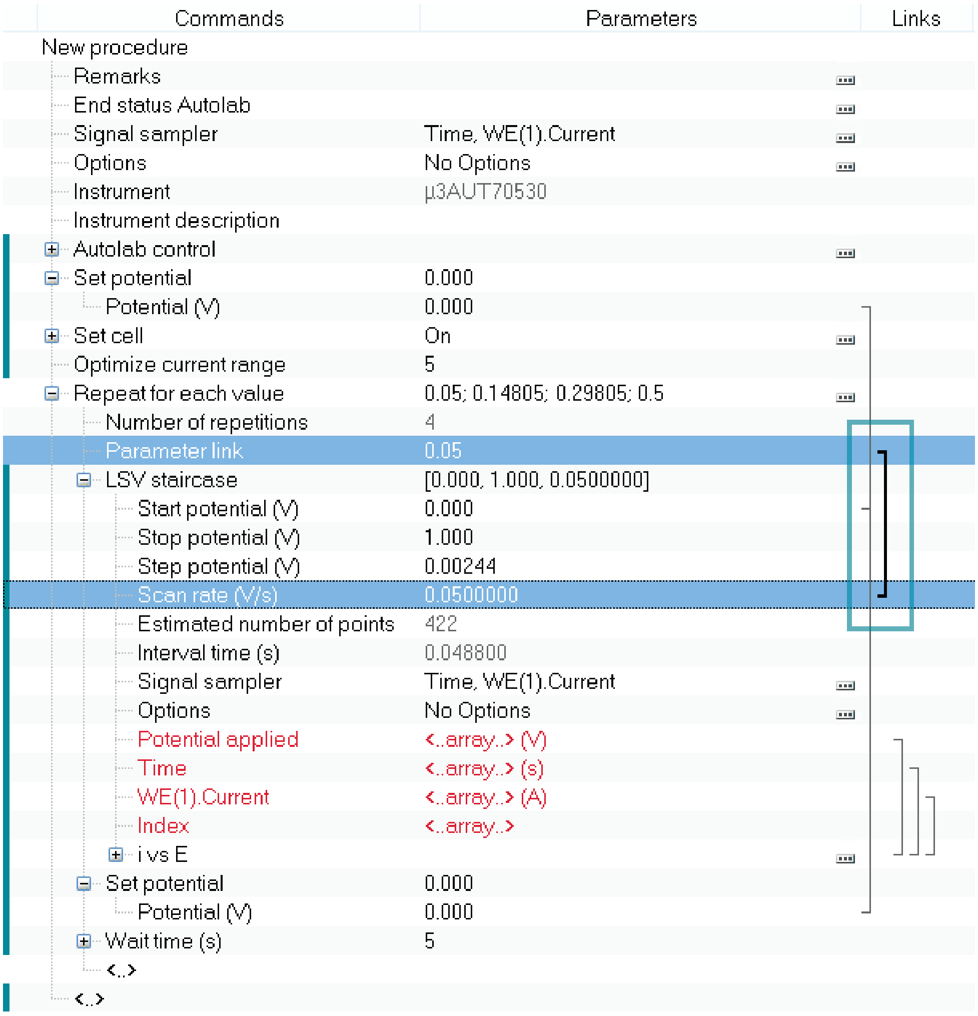


Figure 2.69 – The updated *Repeat for each* value loop: the values are linked to the Scan rate parameter of the *LSV staircase* command

2.5.5 – Defining the post-measurement settings

The final part of this procedure is to switch the cell off after the last linear sweep voltammetry.

For this part of the procedure, a single step is required:

1. Switch the cell off

A *Set cell* command must be added to the procedure. The default parameter setting of the Set cell command is off, which means that the command does not have to be edited.

Figure 2.70 shows the complete procedure, after addition of the final two commands.

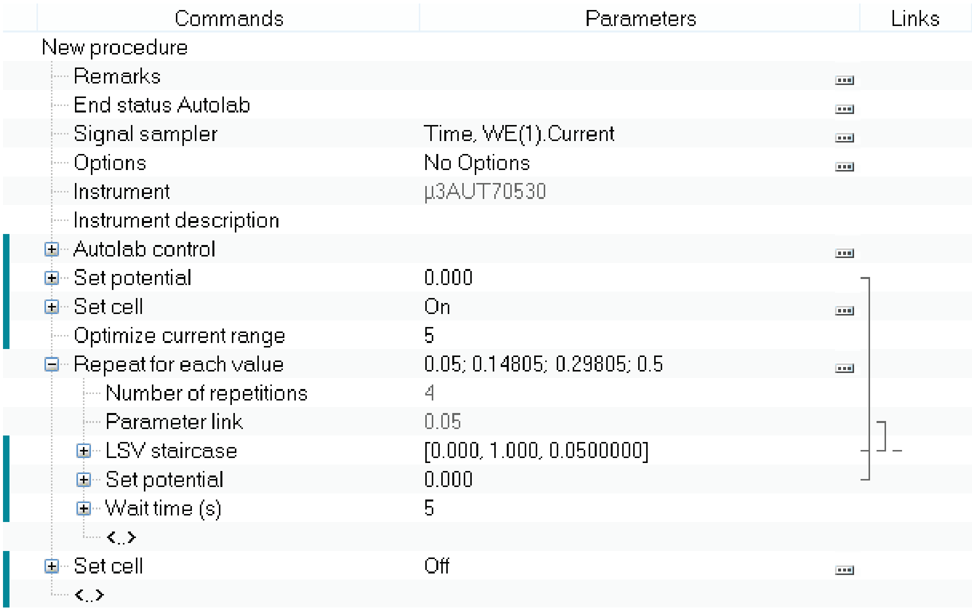


Figure 2.70 – The completed procedure

2.5.6 – Defining the Options

Although this procedure could already be started at this point, it is important to consider the options to be used during the experiment. For this measurement, the Automatic current ranging option will be used.

Click the … button located on the **Options** line of the procedure editor to open the Automatic current ranging window (see Figure 2.71).

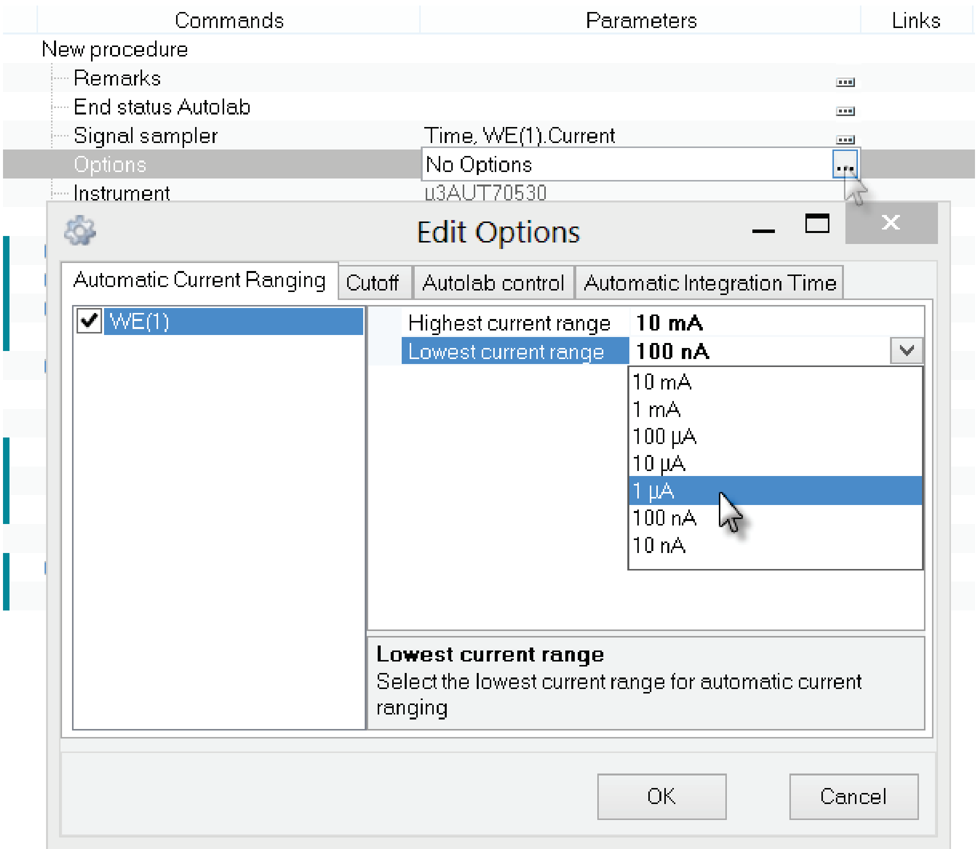


Figure 2.71 – Accessing the Automatic current ranging options

Check the WE(1) checkbox to activate the automatic current ranging option for the working electrode and set the highest and lowest current ranges to 10 mA and 1 µA respectively (see Figure 2.71).

Click the OK button to close the Automatic current ranging window. You will be prompted to define for which measurement commands in this procedure you want to use this option, through the Preview changes window. There are two commands in this procedure that can use the automatic current ranging options (see Figure 2.72). Since these options are intended to be used in the whole procedure, click the Select all button to check all the available measurement command and then press the OK button to validate the options for these commands.

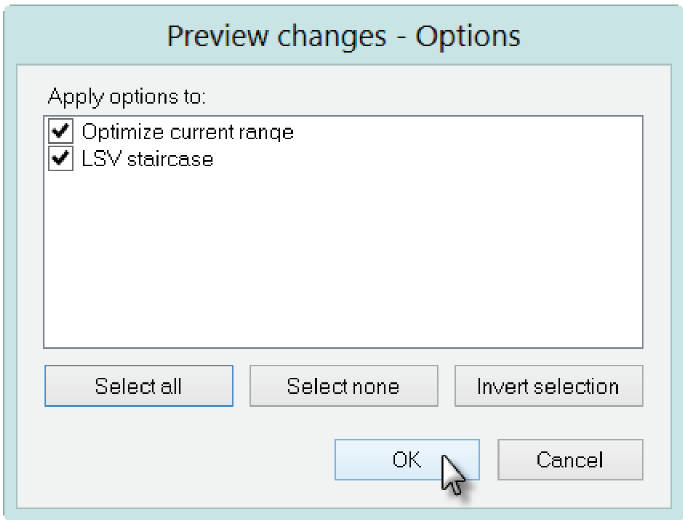


Figure 2.72 – Choosing the measurement commands for which to use the options

Note

It is also possible to open the Options by clicking the button in the quick access toolbar which appears when the procedure header is selected (see Figure 2.73).

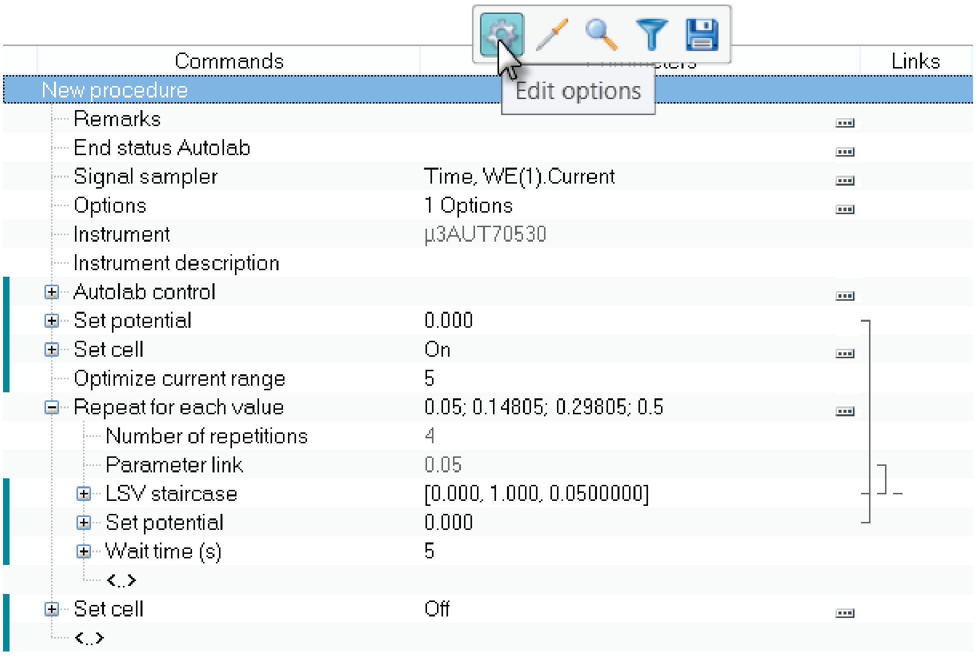


Figure 2.73 – Opening the Options editor can also be done by clicking the button in the quick access toolbar

2.5.7 – Defining the Sampler

It is also important to consider the data acquisition settings (Signal sampler) properly. For this measurement, the potential, current and time will be sampled. The current will be optimized[25].

Click the button located on the **Signal sampler** line of the procedure editor to open the Sampler editor window. Click the WE(1).Potential check box to add the potential to the signal sampler (see Figure 2.74).

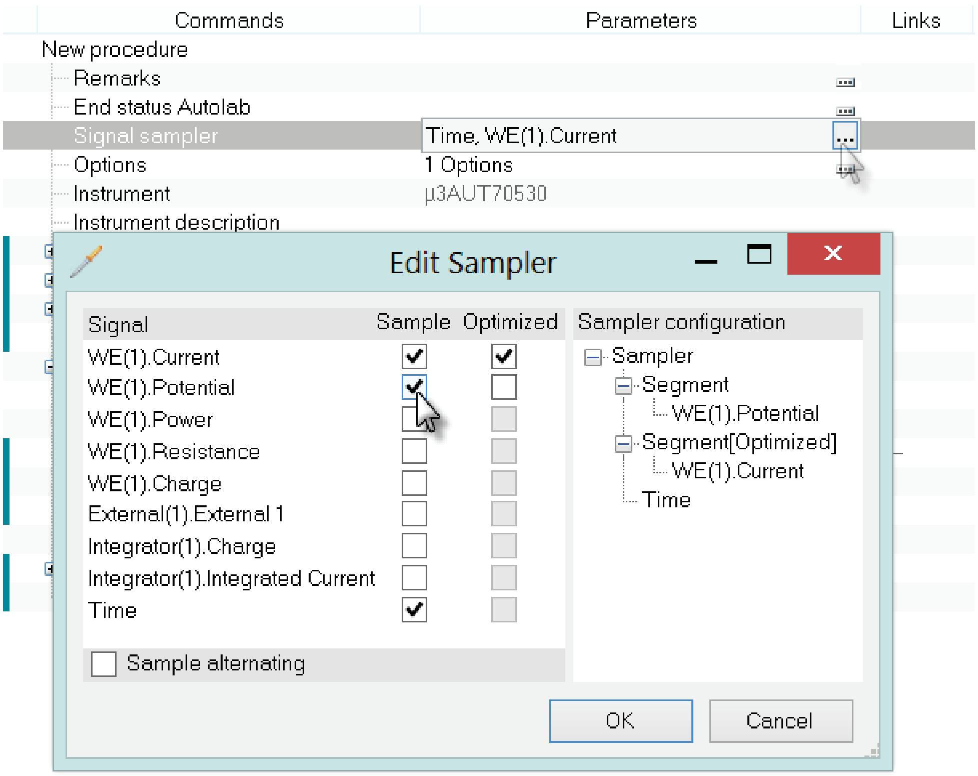


Figure 2.74 – Adding the WE(1).Potential to the sampler

Click the OK button to close the Sampler editor window. Again, NOVA will prompt you to choose for which measurement commands you want to use this sampler, through the Preview changes window (Figure 2.75). Select all the measurement command and press the OK button.

[25] Please refer to Chapter 1 of this User Manual for more information on the different sampling strategies in Nova.

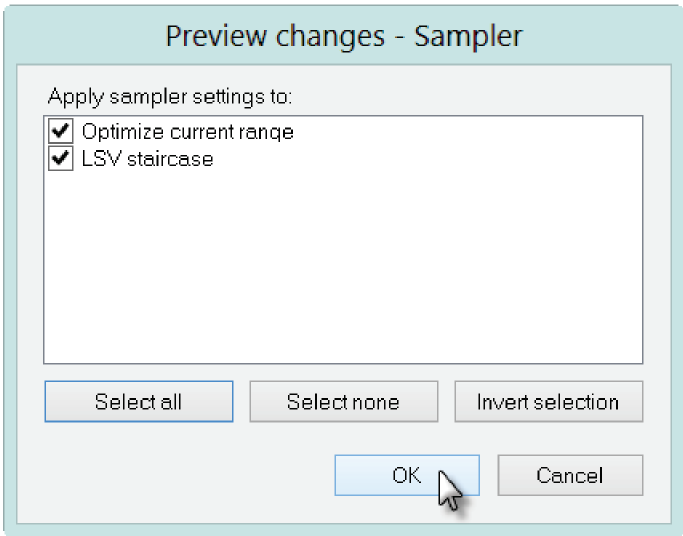


Figure 2.75 – Choosing the measurement commands for which to use the sampler

Note

It is also possible to open the Sampler by clicking the button in the quick access toolbar which appears when the procedure header is selected (see Figure 2.76).

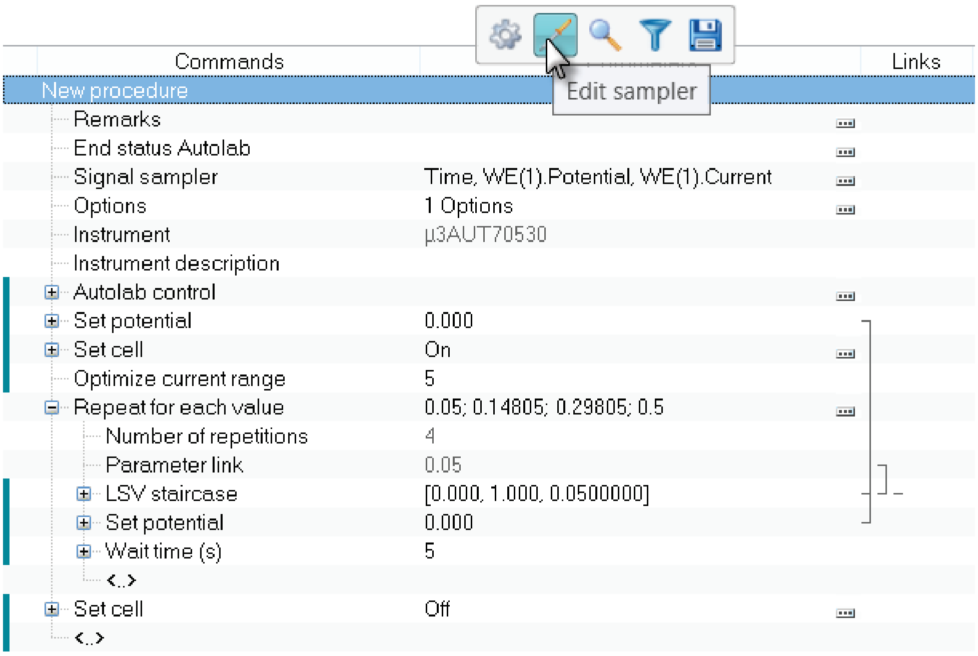


Figure 2.76 – Opening the Sampler editor can also be done by clicking the button in the quick access toolbar

2.5.8 – Defining the plot settings

The procedure setup allows the definition of the plot settings to be used in the measurement view. Each measurement command, like *LSV staircase*, has a signal sampler, which provides a number of signals. These signals can be linked to a *Plot* command, which is attached to the measurement command. By default, the *LSV staircase* command has a pre-defined *Plot* command called **i vs E** (see Figure 2.77).

Plot commands like the **i vs E** plot used in this procedure have two parameters:

- **Show during measurement: Yes/No** – defines whether the plot should be displayed during the measurement or not.
- **Measurement plot number: 1, 2, 3 or 4** – defines the location of the plot in the measurement view. Up to four plots can be shown real-time in the measurement view.

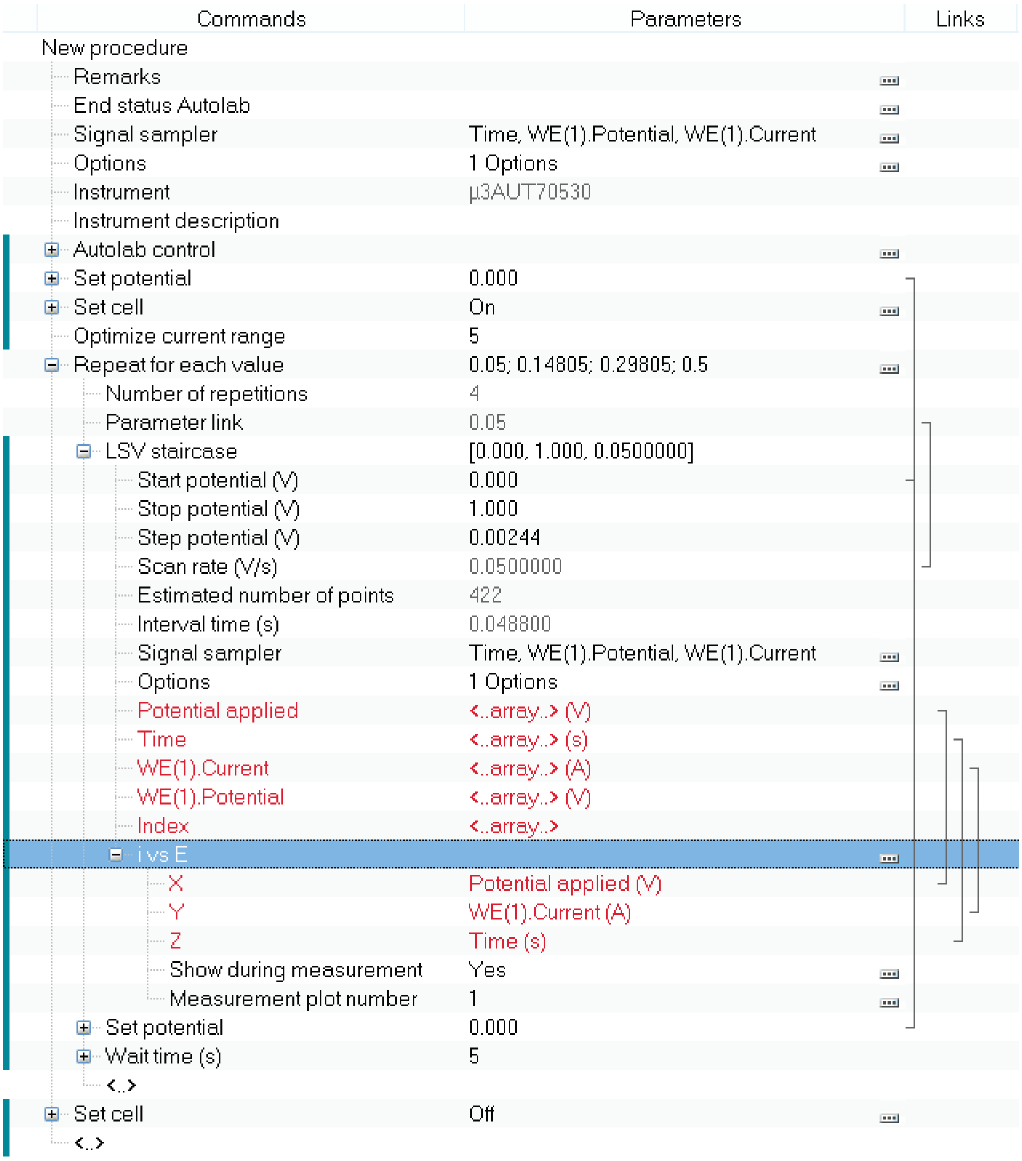


Figure 2.77 – The *i vs E Plot* used in combination the *LSV staircase* command

The plot settings are defined using links. The links are located between the electrochemical signals provided by the *LSV staircase* command (Potential applied, WE(1).Potential, WE(1).Current, Time and Index) and the X, Y and Z of the i vs E command.

Using a link, it is possible to plot any combination of the available electrochemical signals in the measurement view.

It is also possible to create more than one plot in the measurement view. Click the *LSV staircase* command. This triggers the quick access toolbar to appear (see Figure 2.78).

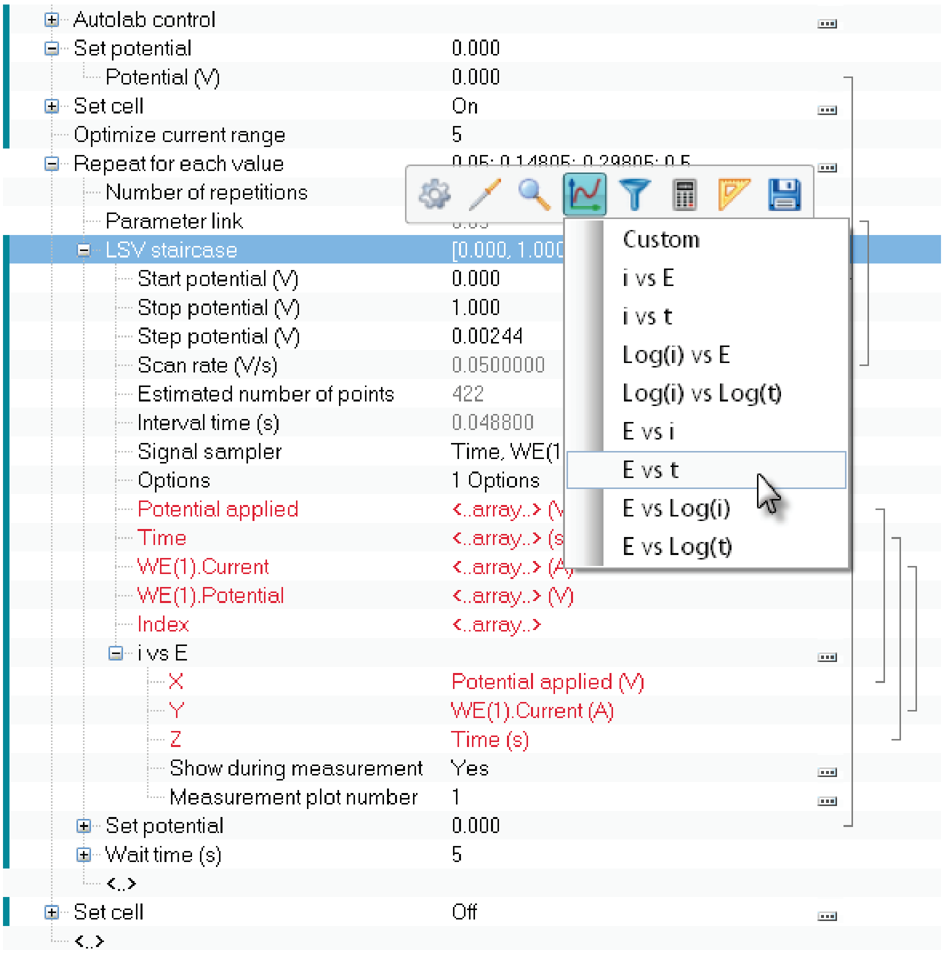


Figure 2.78 – The quick access toolbar can be used to add plots to the measurement command

Note

The list of plots shown in the quick access toolbar depends on the signals defined in the signal sampler. If the WE(1).Potential signal is not sampled, the E vs t and the E vs log(t) plots will not be available (see Figure 2.79).

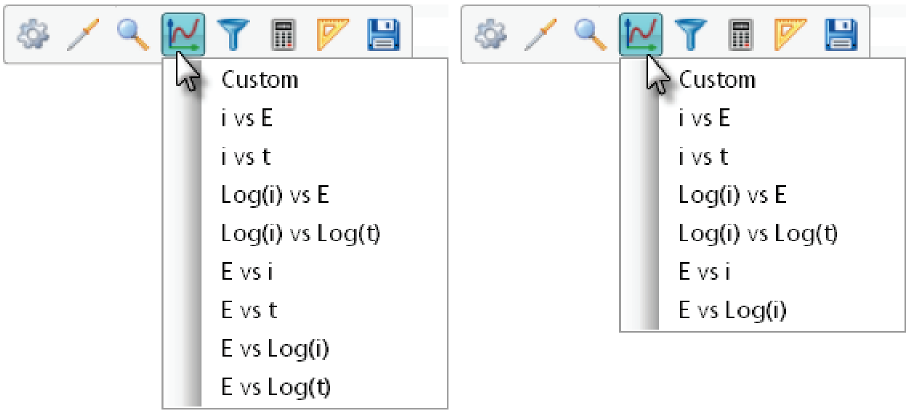


Figure 2.79 – The list of plots shown in the drop-down list depends on the signal sampler settings (left: WE(1).Potential sampled; right: WE(1).Potential not sampled)

Click the  button in this toolbar to display a list of available plots in a drop-down list and select the E vs t plot to add it to the *LSV staircase* command.

The *E vs t* will be added to the *LSV staircase* command and the X and Y inputs of the plot will linked **automatically** to the signals provided by the command (see Figure 2.80).

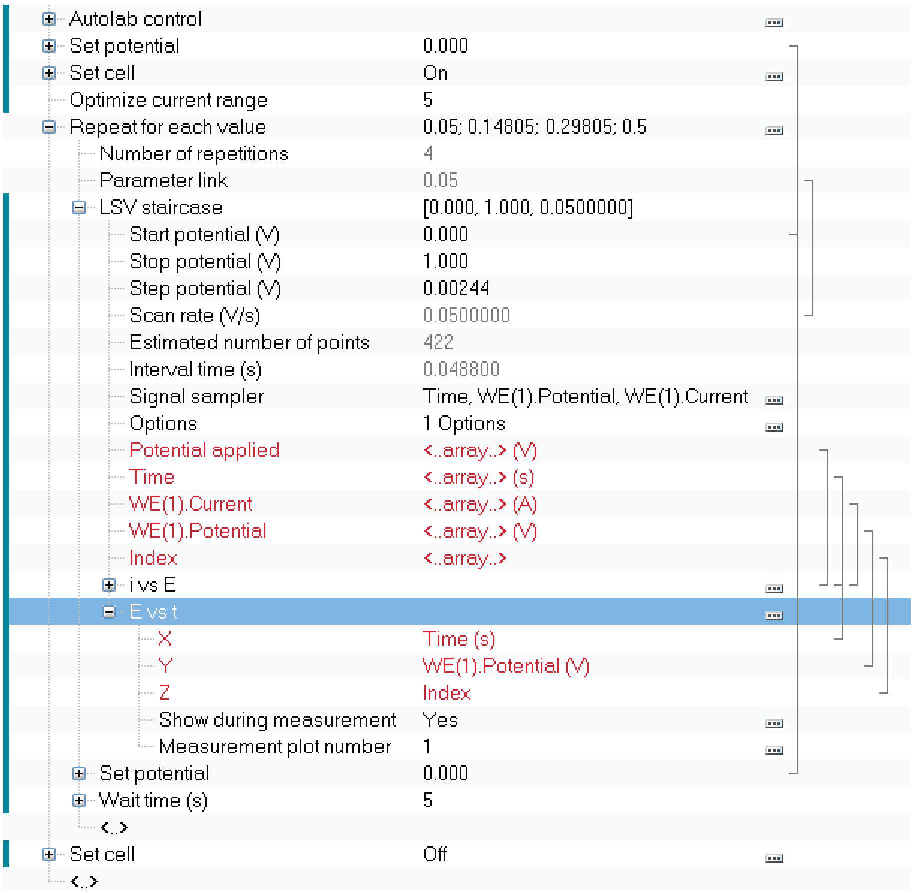


Figure 2.80 – The *E vs t* plot command is automatically linked to the signals provided by the *LSV staircase* command

Note

Plots are automatically linked to the required signals if these signals are specified in the signal sampler. In this case, the WE(1).Potential and the Time signal are both specified in the signal sampler of the *LSV staircase* command and the *E vs t* plot can therefore automatically link its X and Y axis to these signals when the plot is added to the measurement command.

Change the location of the *E vs t* plot command to plot #2 using the drop-down list provided for the Measurement plot number parameter (see Figure 2.81).

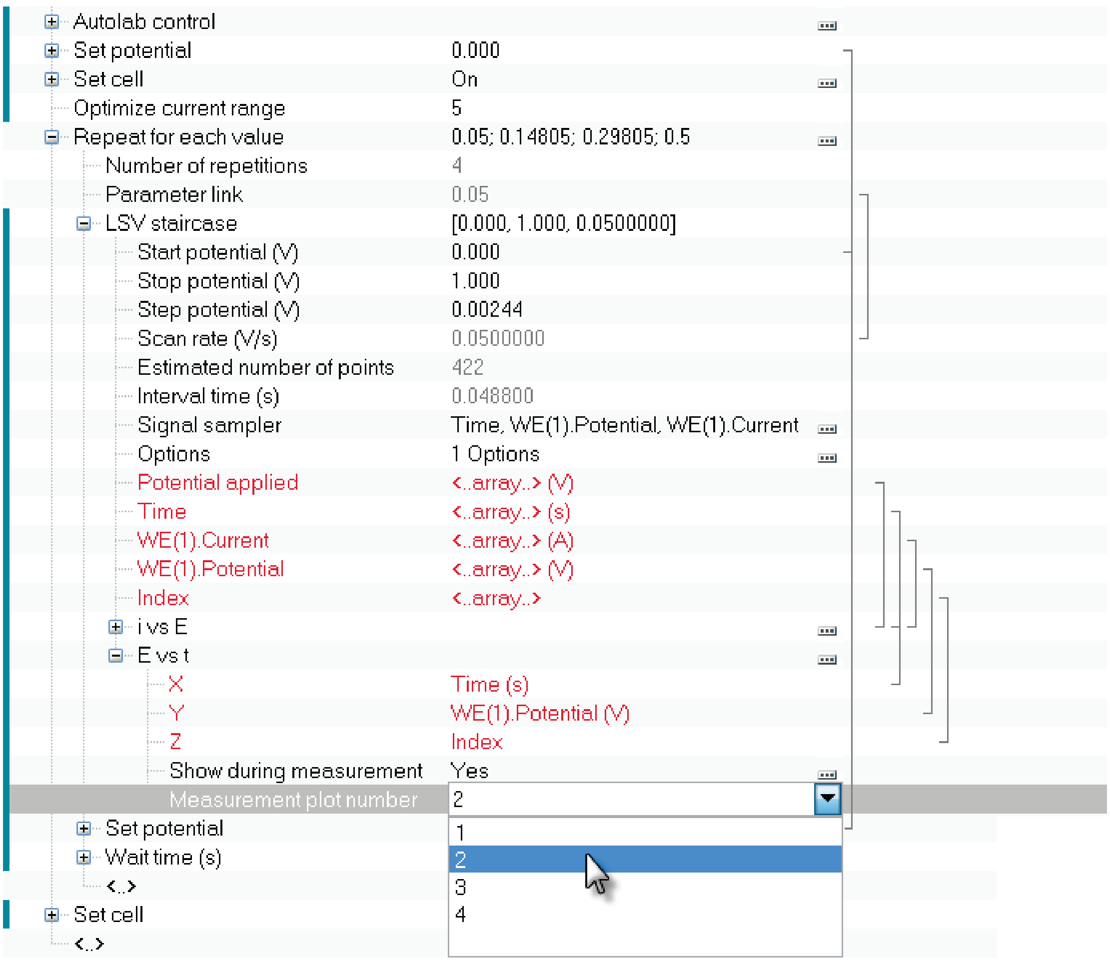


Figure 2.81 – Defining the location of the E vs t plot

2.5.9 – Edit remarks and save procedure

Before running this procedure, it is convenient to use the **Remarks** line at the top of the screen to enter a comment. This will prove useful in the data analysis view and it will help sort the experimental data. To edit the remarks, click the remarks line and click the button displayed right of the Remarks line in the procedure editor. This will open a new window in which you can enter a series of comments (see Figure 2.82).

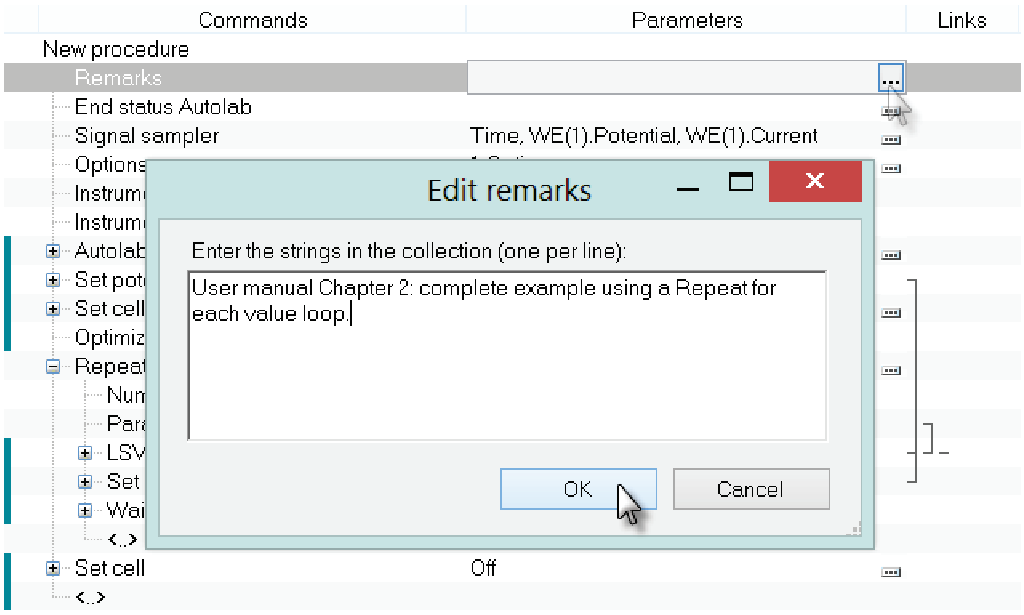


Figure 2.82 – Changing the remarks for the Cyclic voltammetry potentiostatic procedure

The remarks can be used to add information on the cell setup (electrolyte, working electrode, reference electrode, etc.). This information will be stored alongside the measured data.

When the remarks field has been edited, the procedure can be saved into the *My procedures* database. To do this, change the name of the procedure from *New procedure* to *User manual example* in the procedure editor and go to the **File** menu in the toolbar, and select the *Save procedure as new* option. This will add the currently edited procedure to the *My procedures database*. The procedure is logged in this database by name, time and date and, if the mouse pointer is moved over it, it will display a tooltip showing the remarks for this procedure as well (see Figure 2.83).

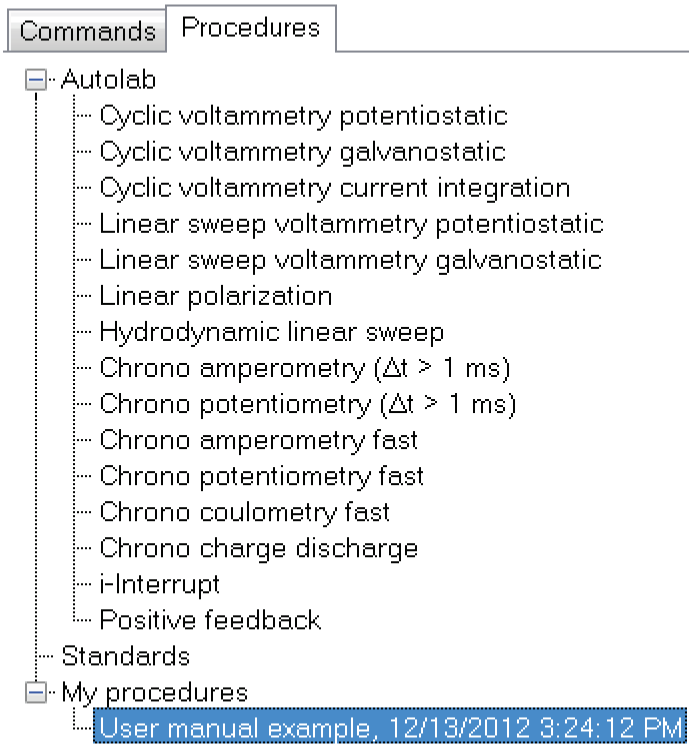


Figure 2.83 – The procedure is added to the My procedures database

Note

The remarks field can contain several lines of comment. All the added lines will be displayed in the tooltip which is very convenient for managing several user defined procedures. The tooltip also displays the location of the procedure file.

2.5.10 – Procedure validation

When the procedure is finished, the validation tool can be used to check for errors. From the Tools menu, select the Check Procedure options (see Figure 2.84).

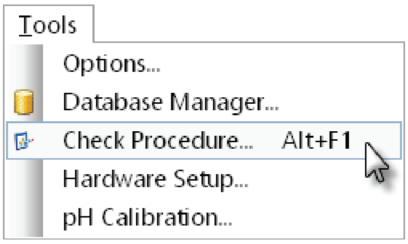


Figure 2.84 – The Check procedure tool is available in the Tools menu

Alternatively the keyboard shortcut Alt + F1 can also be used. NOVA will check the procedure for errors. If no errors are detected, the *No problems found* Message will be displayed (see Figure 2.85).

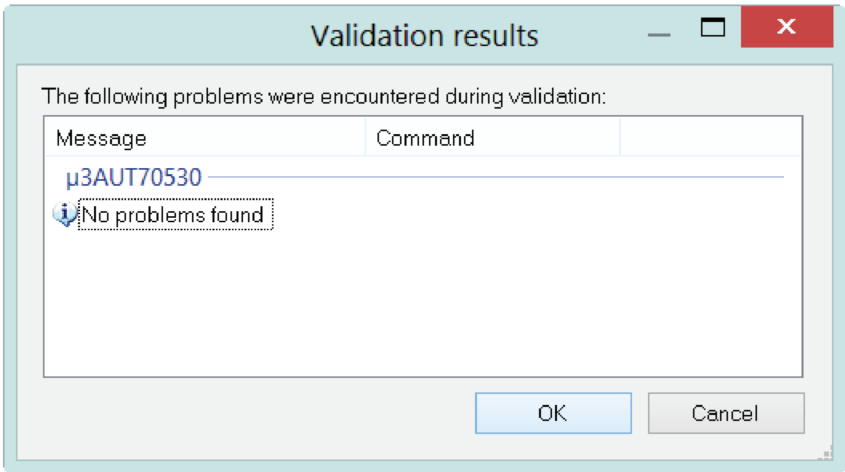


Figure 2.85 – The No problems found message is displayed when no errors are detected

2.5.11 – Running the measurement

Figure 2.86 shows the resulting linear sweep voltammograms obtained using this procedure. The four curves were recorded on one after the other and are available as a single entry in the database. Each curve was obtained for a different scan rate and as the scan rate increases, the peak current also increases.

The curves correspond to the $[Fe(CN)_6]^{4-}/[Fe(CN)_6]^{3-}$ (0.05 M/0.05 M in 0.2 M NaOH) electron transfer on polycrystalline platinum. The reference electrode was a Ag/AgCl (KCl saturated) and the counter electrode was a platinum sheet.

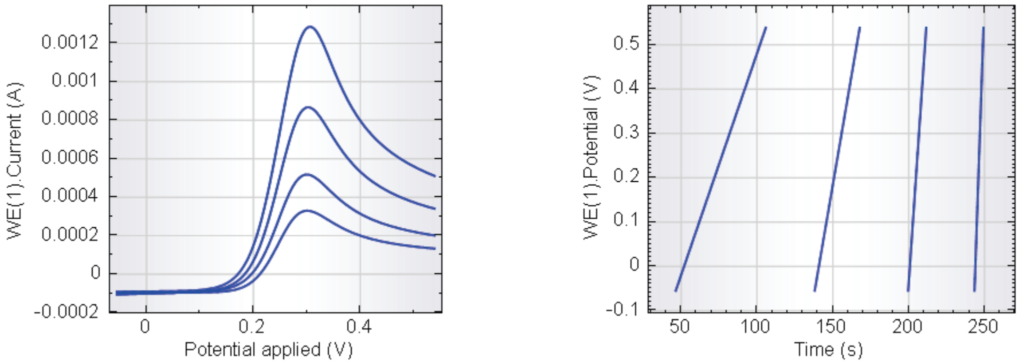


Figure 2.86 – The LSV curves obtained using the procedure described in this section (left – plot #1: i vs E, right – plot #2: E vs t)

2.6 – Dragging and dropping procedures

This section of this chapter provides details on procedure constructions using pre-defined procedures as building blocks. The difference with the previous part of this chapter is that using this editing mode, instead of dragging and dropping commands from the commands browser into the procedure editor, entire procedures will be dragged, from the procedure browser into the procedure editor.

This advanced mode of procedure editing allows for even more flexibility.

Create a new procedure by clicking the new procedure button, , in the toolbar to clear the editor frame. Once the procedure editor has been cleared, it is possible to start building a procedure out of existing procedure.

As an example, a procedure containing the pre-defined Autolab cyclic voltammetry potentiostatic procedure and the Autolab cyclic voltammetry galvanostatic will be constructed.

Using the drag and drop method, select the Cyclic voltammetry procedure and drop it into the procedure editor (see Figure 2.87).

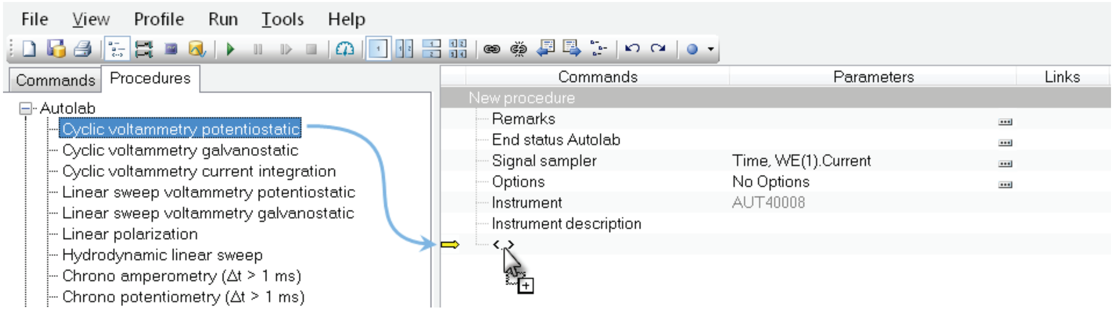


Figure 2.87 – Dragging and dropping the Autolab Cyclic voltammetry potentiostatic procedure into the procedure editor

When the mouse button is released, a message will be displayed, prompting you to choose which Options and Sampler settings to use in the new procedure (see Figure 2.88).

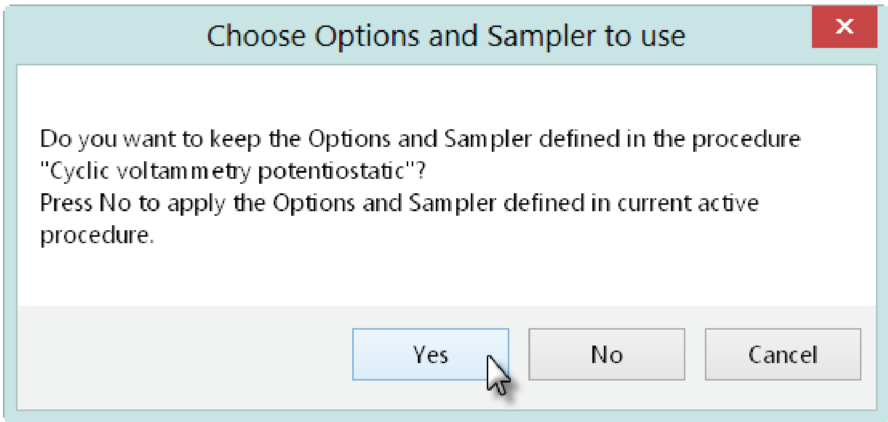
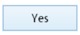


Figure 2.88 – Choosing the Options and Sampler settings to use for the Cyclic voltammetry potentiostatic

The following choices are available:

- **Yes** – the 'Cyclic voltammetry potentiostatic' procedure will be added to the current procedure in the editor frame. The Options and Sampler settings defined in the Cyclic voltammetry potentiostatic procedure will be used during the measurement (Automatic current ranging from 1 mA to 100 nA, Sampler: WE(1).Current and Time).
- **No** – the 'Cyclic voltammetry potentiostatic' procedure will be added to the current procedure in the editor frame. The Options and Sampler currently defined in the procedure editor will be used (No automatic current ranging, Sampler: WE(1).Current and Time). The specific Options and Sampler defined in the Cyclic voltammetry potentiostatic procedure will be overridden.
- **Cancel** – the procedure is not added to the procedure editor.

Press the 

button to add the complete procedure to the procedure editor (see Figure 2.89). The Options and Sampler settings defined in the Cyclic voltammetry potentiostatic procedure will be kept.

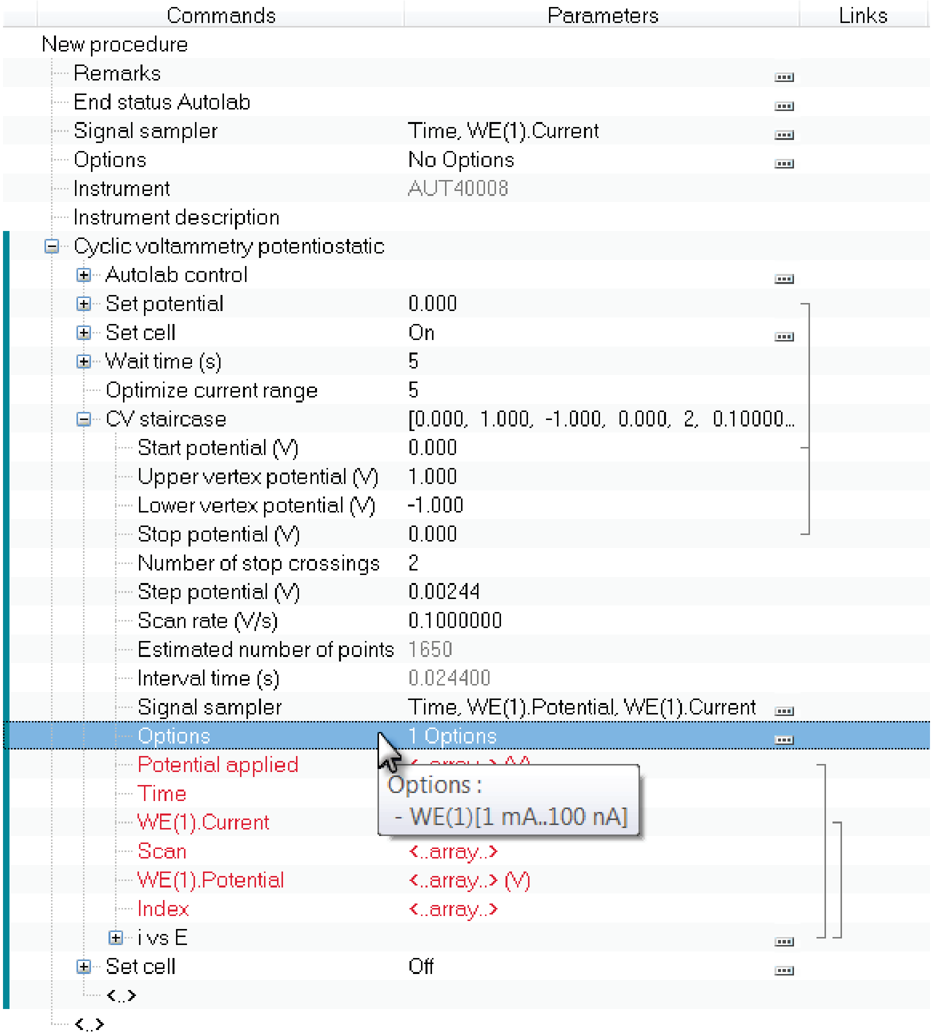


Figure 2.89 – The Cyclic voltammetry procedure added to the procedure editor

Select the Cyclic voltammetry galvanostatic procedure and drop it into the procedure editor, after the cyclic voltammetry procedure. When the mouse button is released, a message will be displayed (see Figure 2.90).

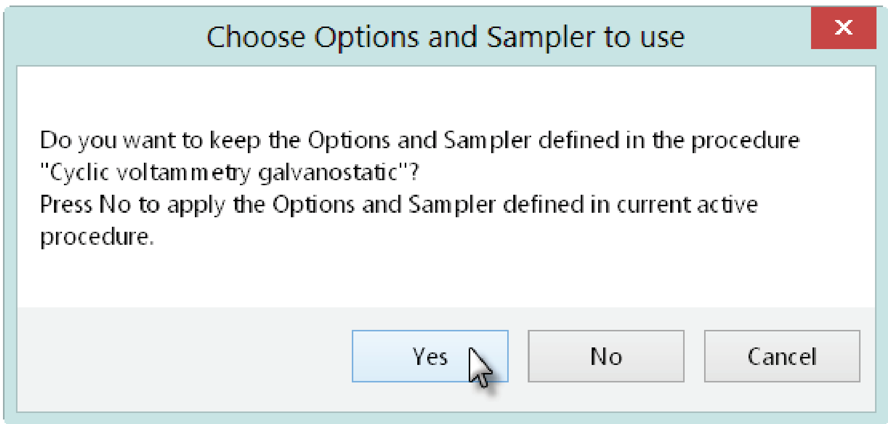


Figure 2.90 – Choosing the Options and Sampler settings to use for the Cyclic voltammetry galvanostatic procedure

Click Yes to continue. The entire Cyclic voltammetry galvanostatic will be added into the procedure editor, just after the Cyclic voltammetry potentiostatic procedure (see Figure 2.91). The Options and Sampler defined in the Cyclic voltammetry galvanostatic procedure will be used.

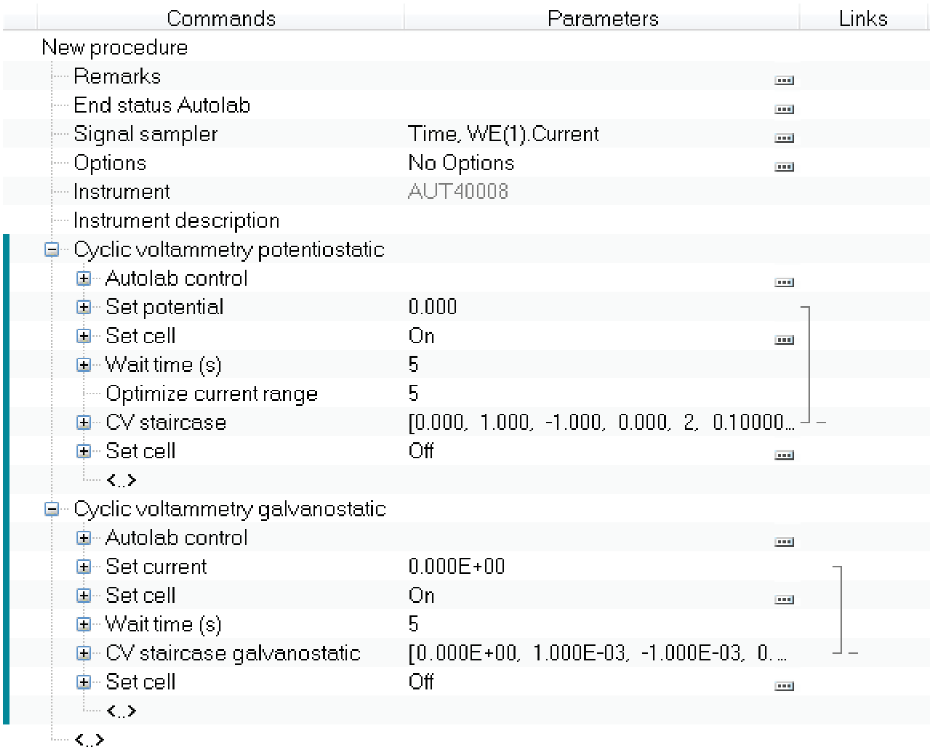


Figure 2.91 – The procedure editor with the two complete procedures

Connect dummy cell (c) and press the start button. When the measurement starts, the procedure will first execute the complete Cyclic voltammetry potentiostatic procedure, using Automatic current ranging. When the first part of the measurement is finished, the Cyclic voltammetry galvanostatic procedure starts. The Autolab control command will switch the potentiostat to galvanostatic mode and set the current range to 1 mA, before performing the current scan.

Figure 2.92 and Figure 2.93 show the measured data on the dummy cell (c). The first part of the measurement corresponds to the potentiostatic cyclic voltammetry. The measurement stops after 47 seconds. The second part of the measurement corresponds to the galvanostatic cyclic voltammetry, which starts after 55 seconds. The time difference between the two measurements stems from the preconditioning stage at the beginning of the cyclic voltammetry galvanostatic measurement.

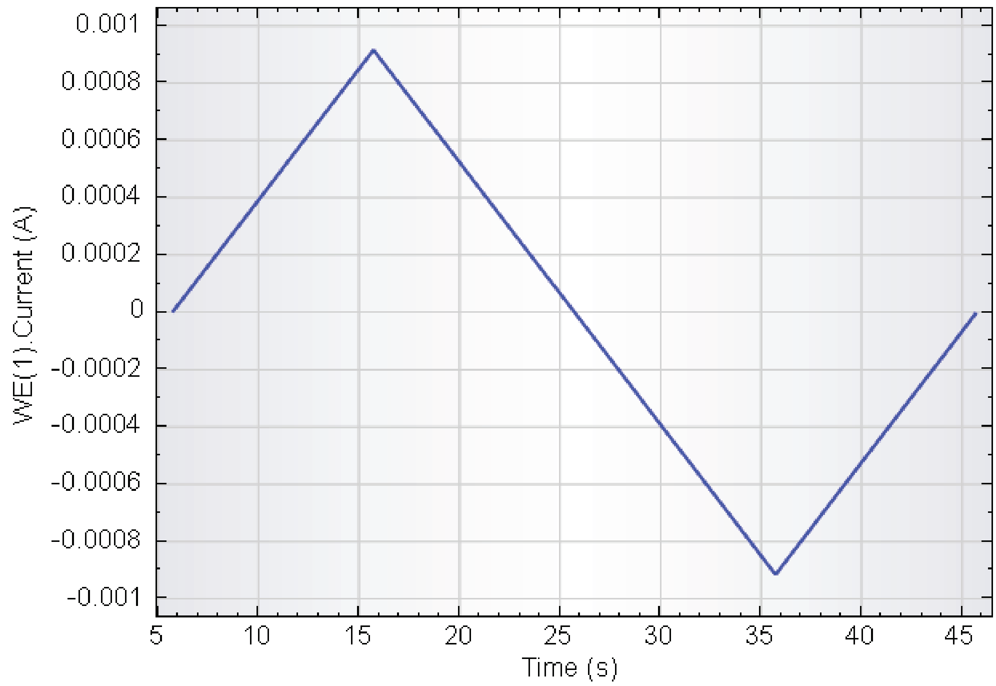


Figure 2.92 – The first part of the measurement (Cyclic voltammetry potentiostatic): measured current vs time

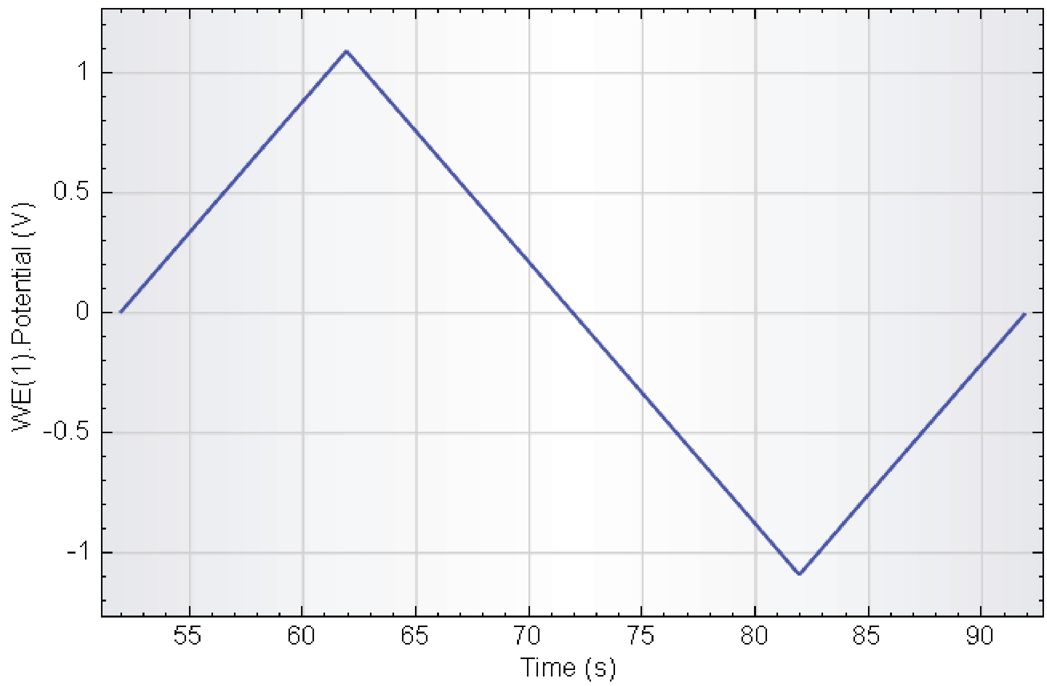


Figure 2.93 – The second part of the measurement (Cyclic voltammetry galvanostatic): measured potential vs time

The Options and Sampler can be modified for the whole procedure or for each individual procedure in the procedure editor. Click the ⋯ button located next the Sampler in the procedure editor or use the quick access toolbar (see Figure 2.94).

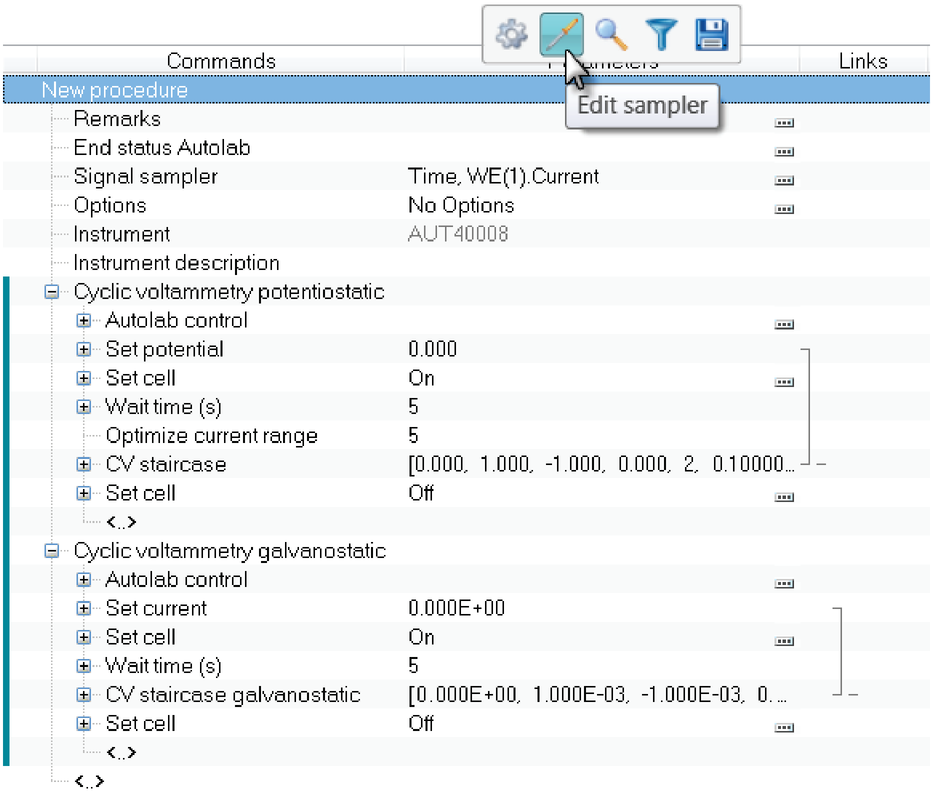


Figure 2.94 – Opening the Sampler for the whole procedure

The sampler editor will be displayed. For illustration purposes, the WE(1).Charge signal will be added to the sampler (see Figure 2.95).

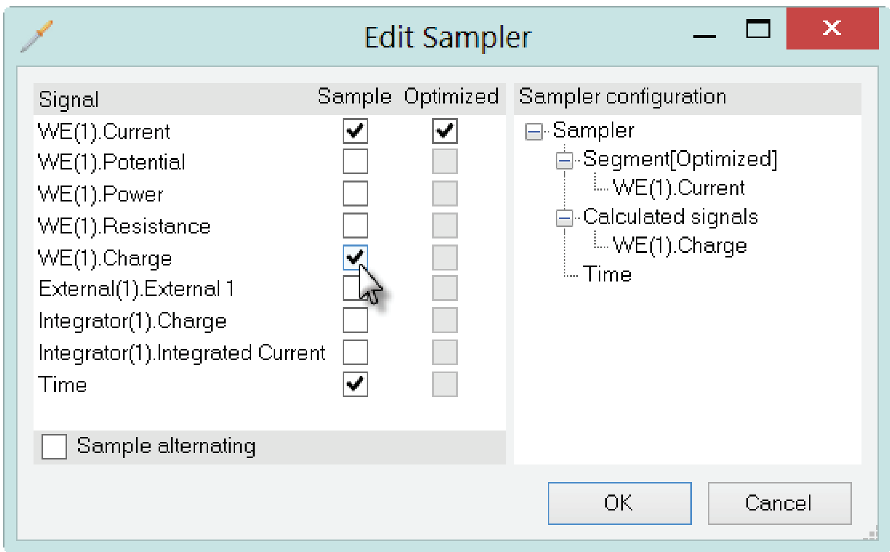


Figure 2.95 – Adding the WE(1).Charge to the Sampler

Click the OK button to close the Sampler window. The **Preview changes** window will be displayed, allowing you to choose for which measurement commands in the procedure this sampler should be used (see Figure 2.96).

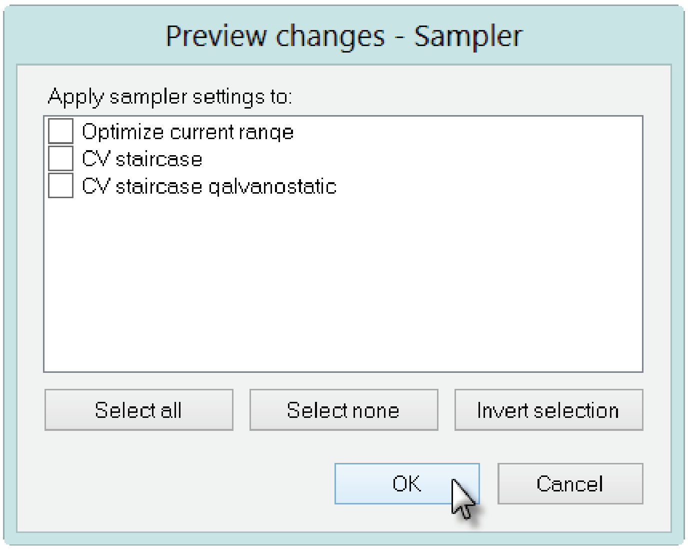


Figure 2.96 – The Preview changes window

2.7 – My commands

The final section of this chapter describes the *My commands* framework. This feature allows you to create new commands. A specific database is available to store your own commands. The location of this database can be defined using the database manager (see Figure 2.97).

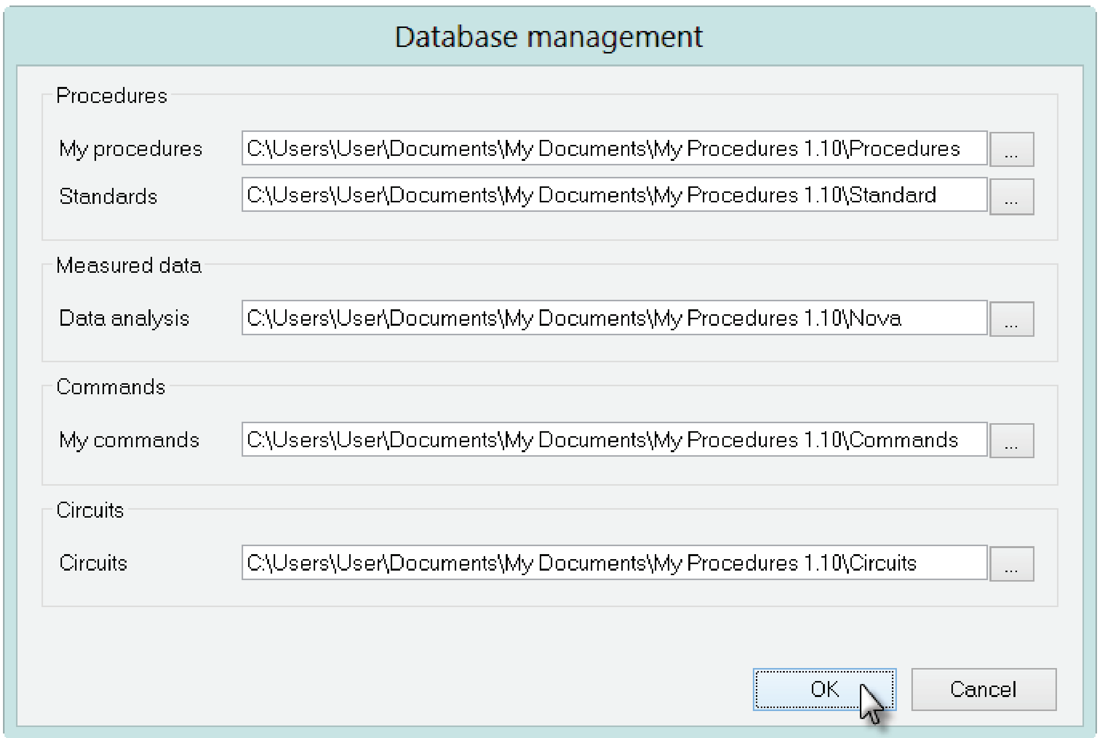


Figure 2.97 – The database manager can be used to define a Commands database

Commands defined using the *My commands* framework will be stored in the defined database and will be available for procedure building in the commands browser (see Figure 2.98).

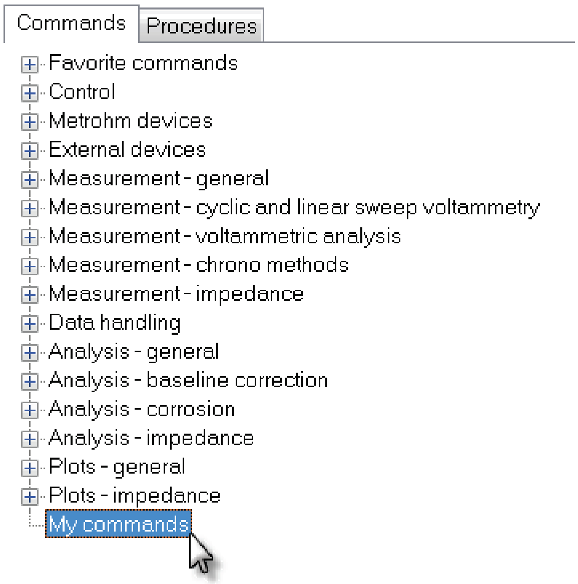


Figure 2.98 – The My commands group is located in the commands browser

2.7.1 – Creating simple My commands

This section illustrates the use of the *My commands* framework in NOVA to create a dedicated single My command. Create a new procedure by clicking the new procedure button, , in the toolbar to clear the editor frame.

As an example, a dedicated command to set the Autolab to galvanostic mode and set the current range to 10 mA will be created.

All instrumental settings are defined using the *Autolab control* command. Using the drag and drop method, add a *Autolab control* command to the empty procedure (see Figure 2.99).

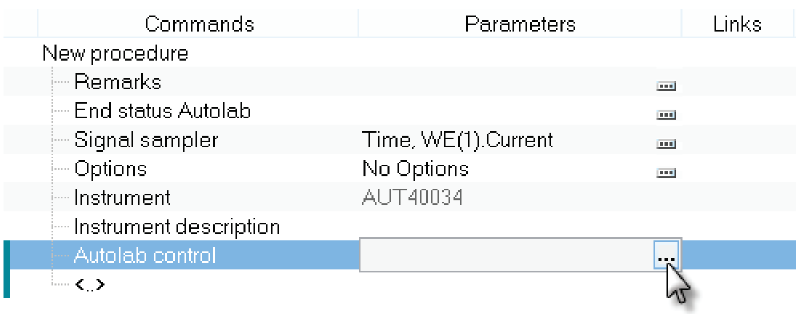


Figure 2.99 – Adding a Autolab control command

Click the ... button to open the Autolab control editor window, as shown in Figure 2.99. In the Autolab control window, adjust the settings on the main potentiostat/galvanostat section (see Figure 2.100).

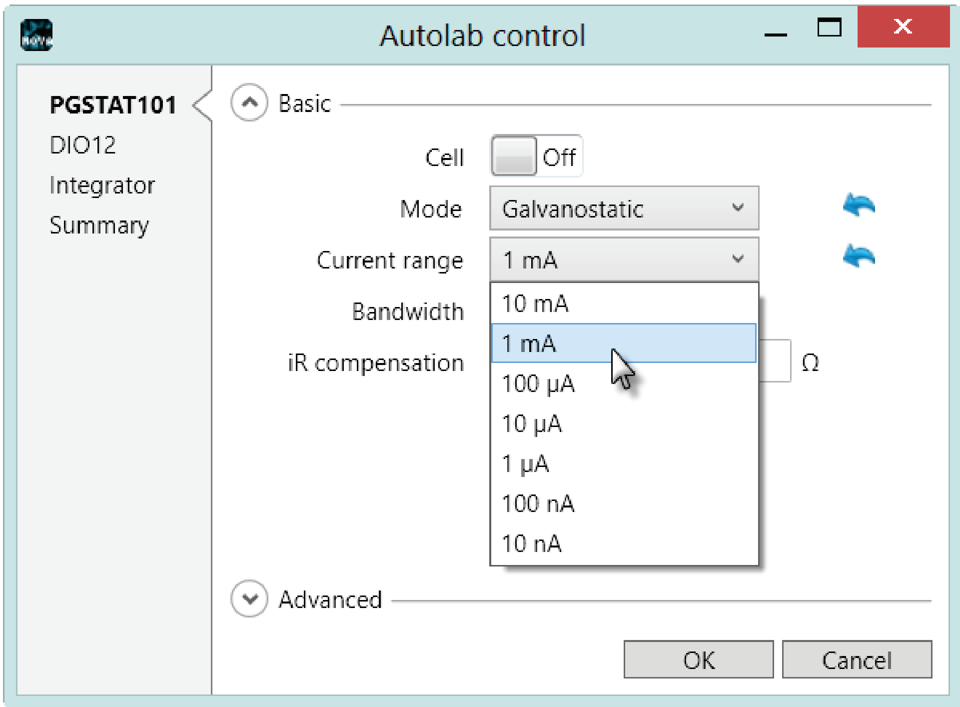


Figure 2.100 – Adjust the settings in the *Autolab control* command

Click the OK button to close the editor. The specified settings will be displayed in the procedure editor (see Figure 2.101).

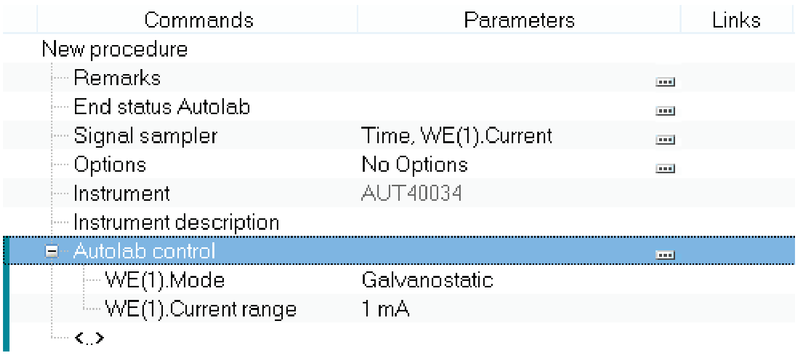


Figure 2.101 – Settings defined in the Autolab control command are shown in the procedure editor

To save the *Autolab control* command with all the defined settings in the My commands group, right-click on the *Autolab control* command and select the Save in My commands option from the context menu (see Figure 2.102).

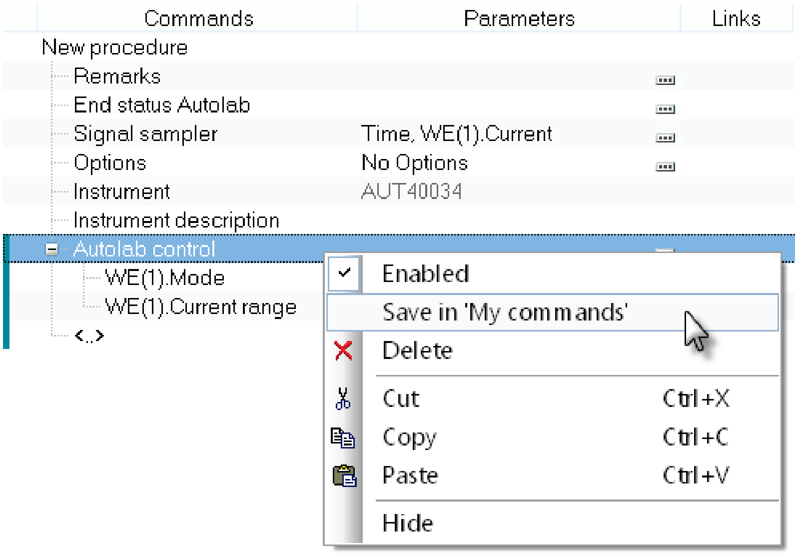


Figure 2.102 – Right-click the *Autolab control* command and select the Save in My commands option

Aternatively, it is also possible to click the button in the quick access toolbar (see Figure 2.103).

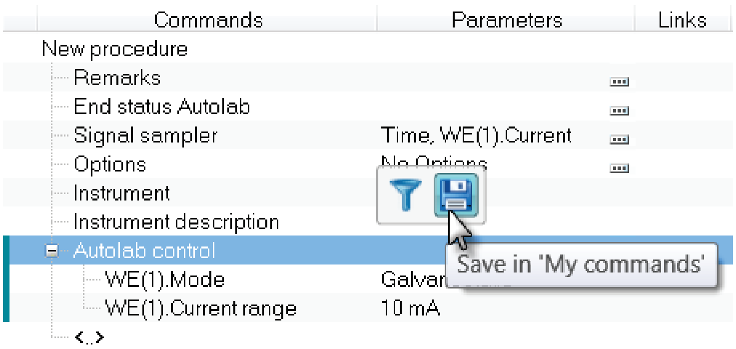


Figure 2.103 – Click the icon in the quick access toolbar to save the *Autolab control* command

A window will be displayed. Herein, a name for the My command can be specified and remarks can be added. This is helpful for bookkeeping purposes, in order to identify the command more easily. For this example, a name and remarks are provided as shown in Figure 2.104.

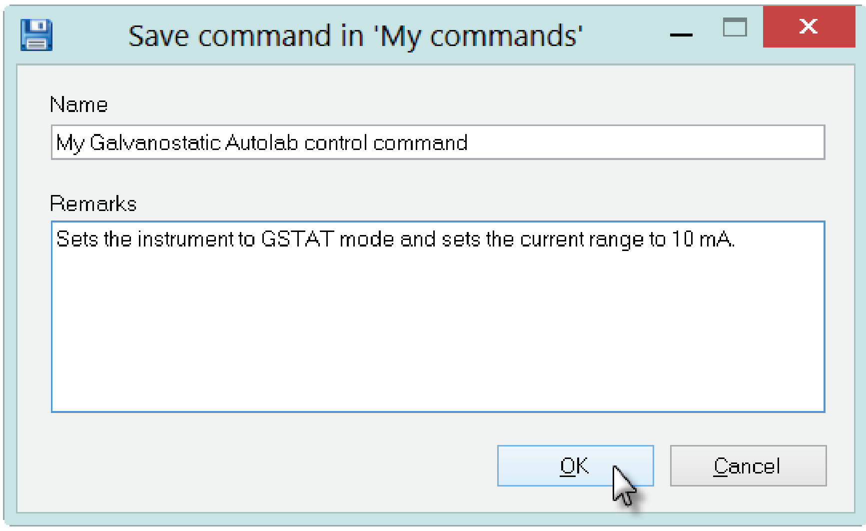


Figure 2.104 – Defining a name and remarks for the My command

Click the OK button to close the window. This specific version of the Autolab control command will be added to the list of My commands (see Figure 2.105).

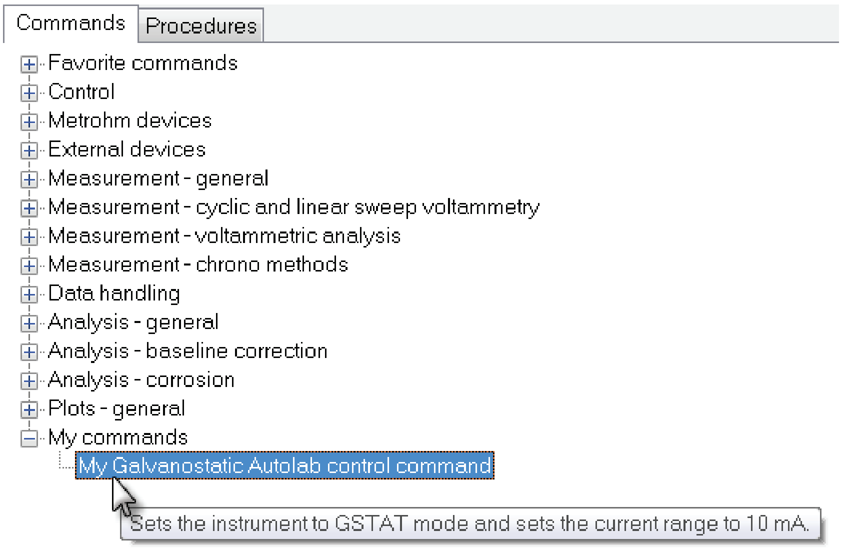


Figure 2.105 – The command is displayed in the My commands list

Note

The remarks are shown in the tooltip.

2.7.2 – Creating complex My commands

The example shown in the previous section shows how to create a single My command. It is also possible to group commands together to create a complex set of commands as a single My command.

For this purpose, a dedicated grouping command, called *Nested procedure*, is used. The Nested procedure command can be found in the Control group of commands (see Figure 2.106).

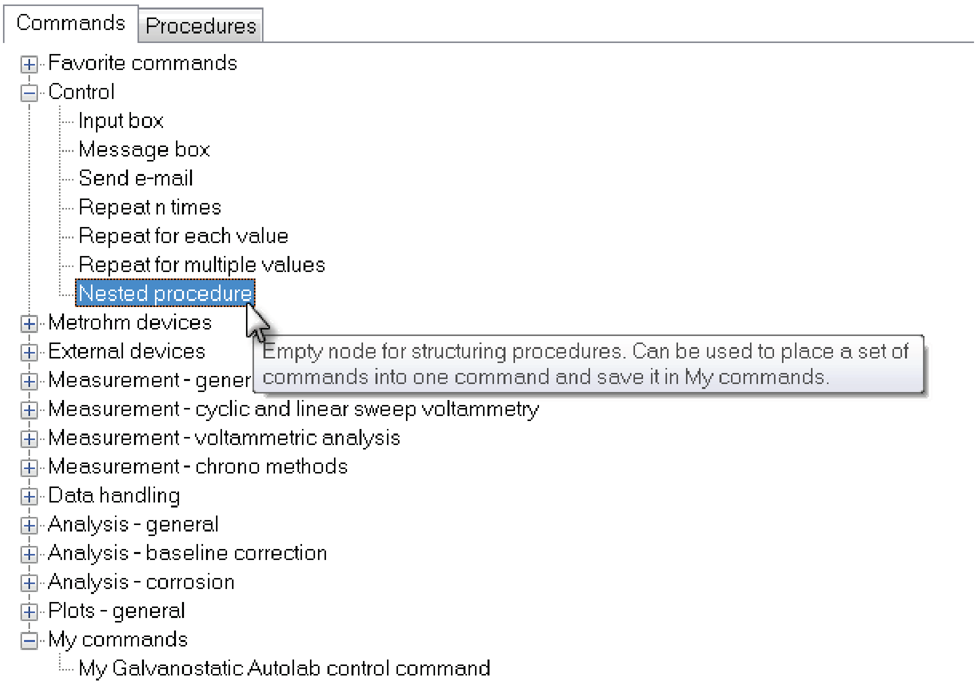


Figure 2.106 – The *Nested procedure* command can be used to group commands

Using a *Nested procedure* command, it is possible to group commands together and save them into the My commands group.

Create a new procedure by clicking the new procedure button, , in the toolbar to clear the editor frame.

As an example, a pre-conditioning command that sets the Autolab, applies 1.2 V, switches the cell ON, waits during 5 seconds and selects the best possible current range will be constructed.

To start, locate the Nested command in the Control group of commands and add it to the procedure editor (see Figure 2.107).

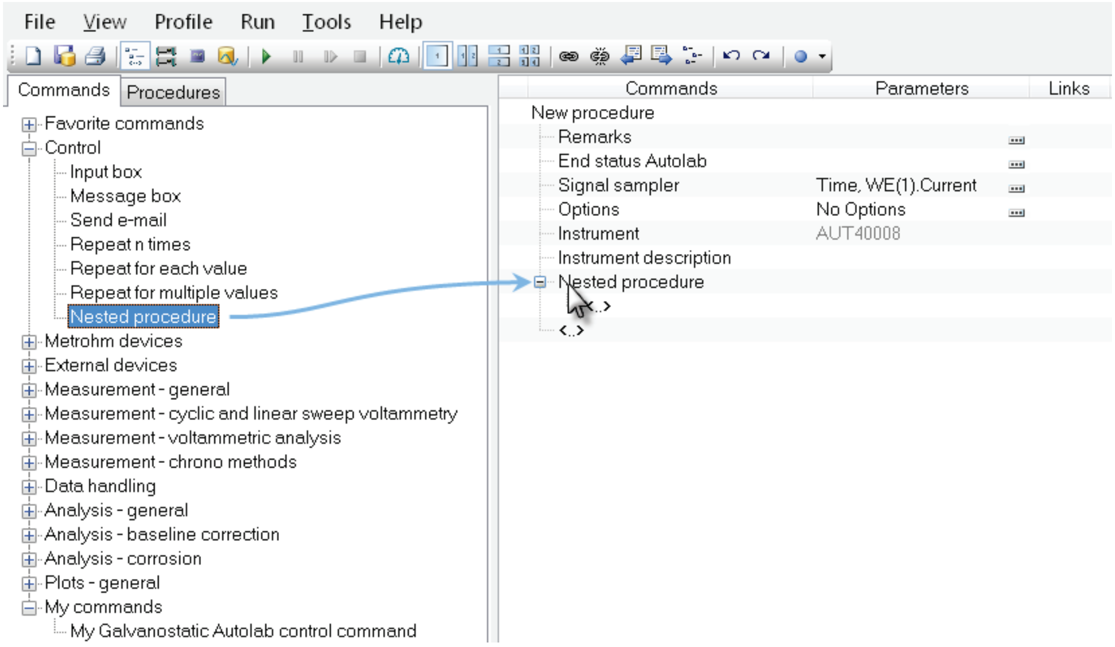


Figure 2.107 – Adding the *Nested procedure* command

The *Nested procedure* command creates a subsequence in the procedure editor (see Figure 2.108). Using the drag and drop method is it possible to add commands to the main sequence or the subsequence created by the *Nexted procedure* command.

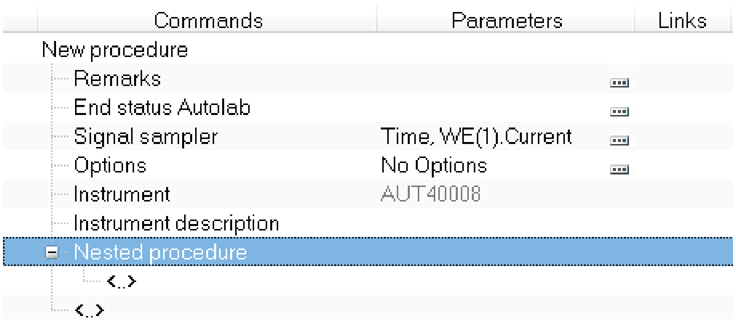


Figure 2.108 – The *Nested procedure* command creates a subsequence in the procedure editor

From the command browser, add the following commands into the procedure editor, ensuring that they are added to the *Nested procedure* sequence (see Figure 2.109):

- Autolab control
- Set potential: 1.2 V
- Set cell On
- Wait time (s): 5
- Optimize current range

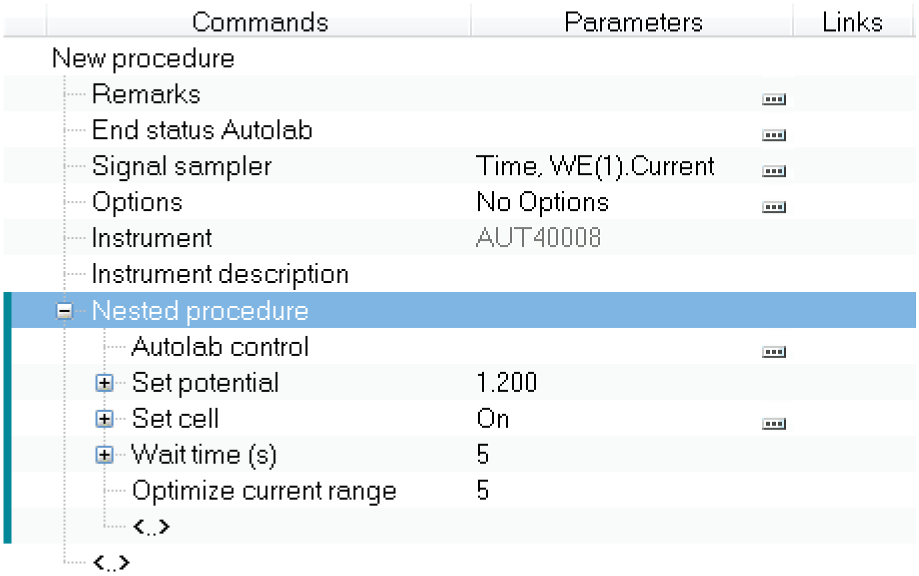


Figure 2.109 – Adding the commands to the procedure editor

Note

Make sure that the commands are added to the Nested procedure sequence.

Use the *Autolab control* command to set the instrument to High stability, select the 1 mA current range and set the Autolab to potentiostatic mode (see Figure 2.110).

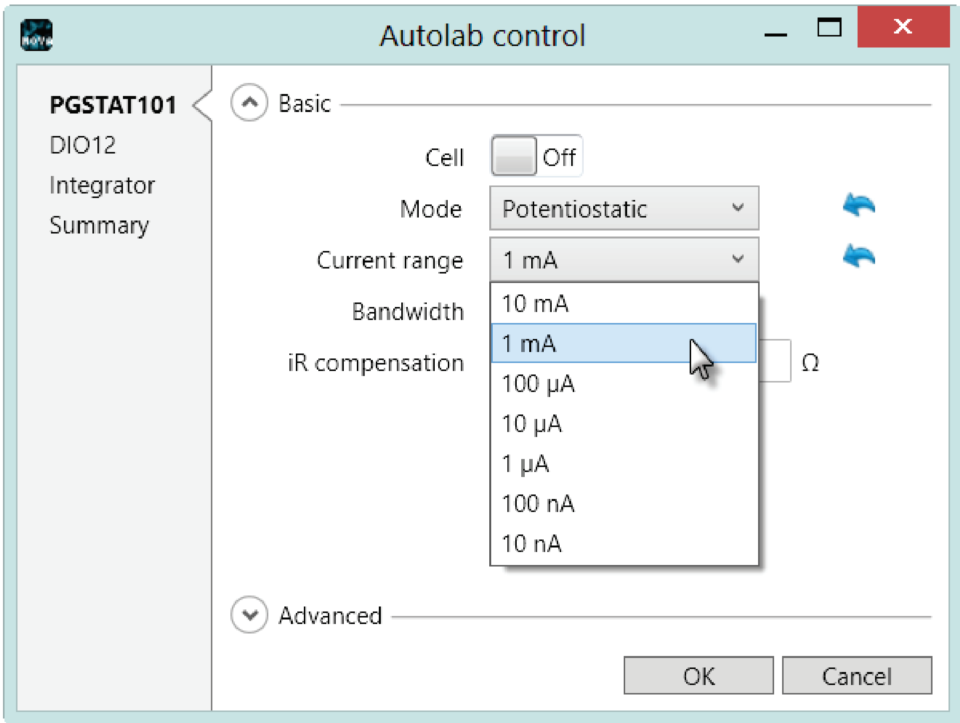


Figure 2.110 – Set the initial Autolab conditions using the Autolab control interface

2.7.3 – Saving My Commands

Right-click the *Nested procedure* command and select the 'Save in My commands' option from the context menu, or use the quick access shortcut icon (see Figure 2.111).

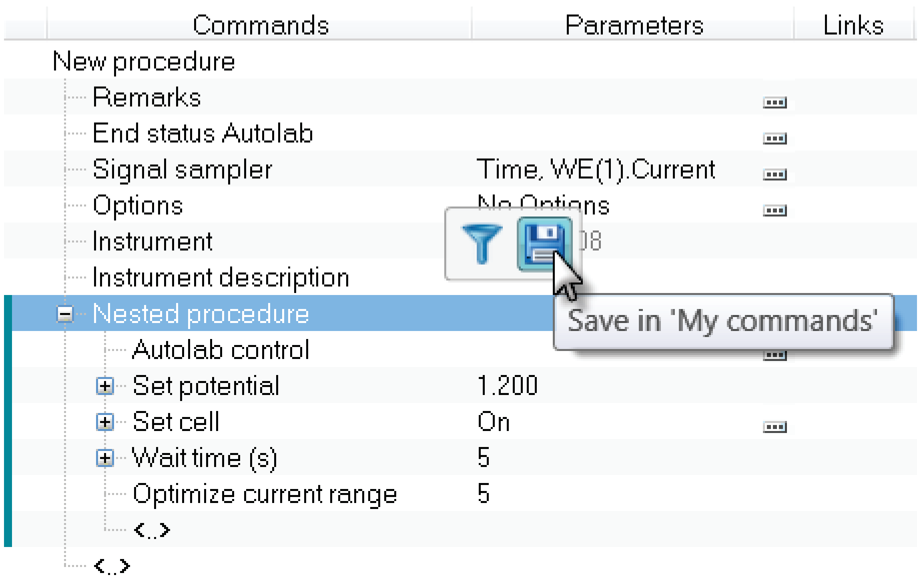


Figure 2.111 – Select the Save in 'My commands' option from the right-click menu

You will be prompted to specify a name and remarks for the command (see Figure 2.112).

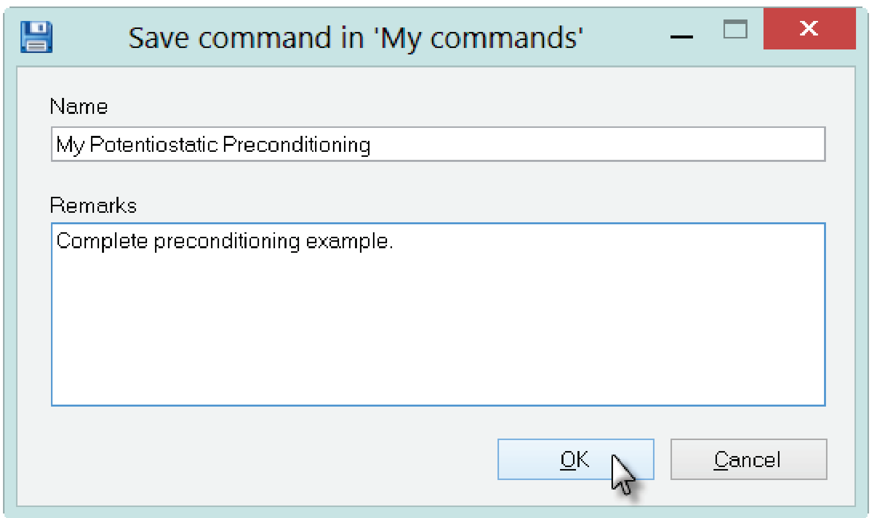


Figure 2.112 – Specify a name and remarks for the command

In the example shown here, the whole *Nested procedure* will be saved in the My commands database, as My Potentiostatic Preconditioning. Once the OK button is clicked, the whole *Nested* procedure will be saved in the My commands database (see Figure 2.113).

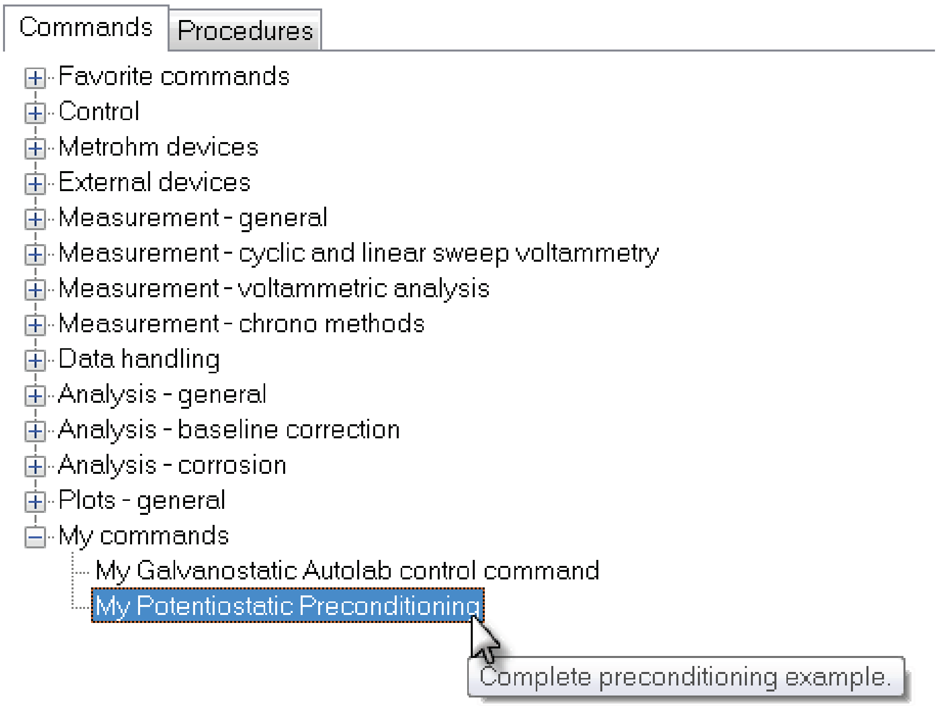


Figure 2.113 – The command is added to the My commands database

2.7.4 – Using the My command

Once a command has been added to the *My commands* database, it can be dragged into the procedure editor as any other command.

Create a new procedure and drag the My Potentiostatic Preconditioning command into the procedure editor. An exact copy of the original command will be added to the procedure (see Figure 2.114).

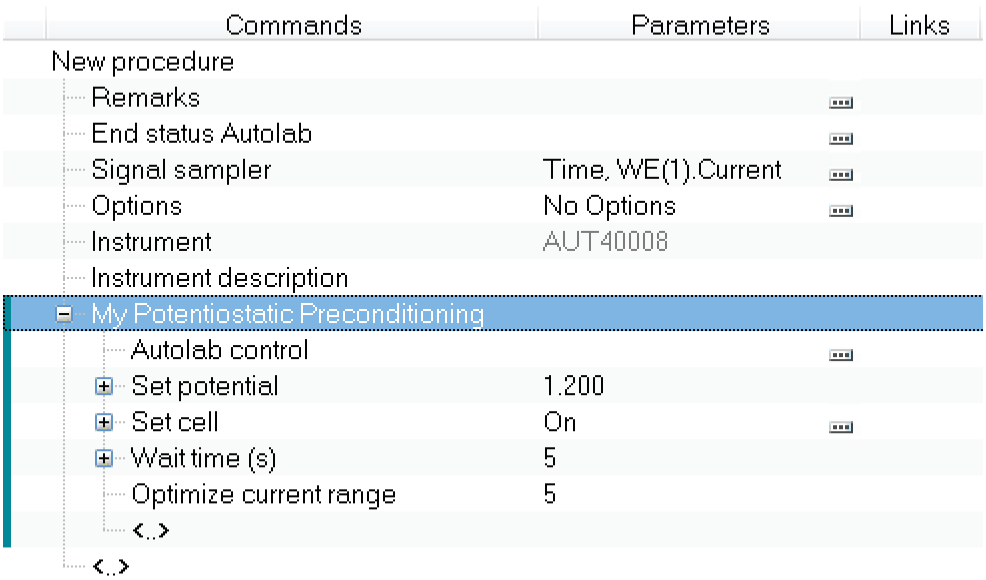


Figure 2.114 – Adding a My command into the procedure editor creates a copy of the original command

Note

Saving a complete *Nested procedure* instead of a single command allows you to very quickly group commands. This simplifies the procedure building process, especially for routine measurements.

3 – The Measurement view

While the procedure is constructed within the setup view, the measured data can be observed in real time using the **Measurement view**. To switch this view, click the corresponding button in the toolbar or select the measurement view option from the View menu (Figure 3.1).

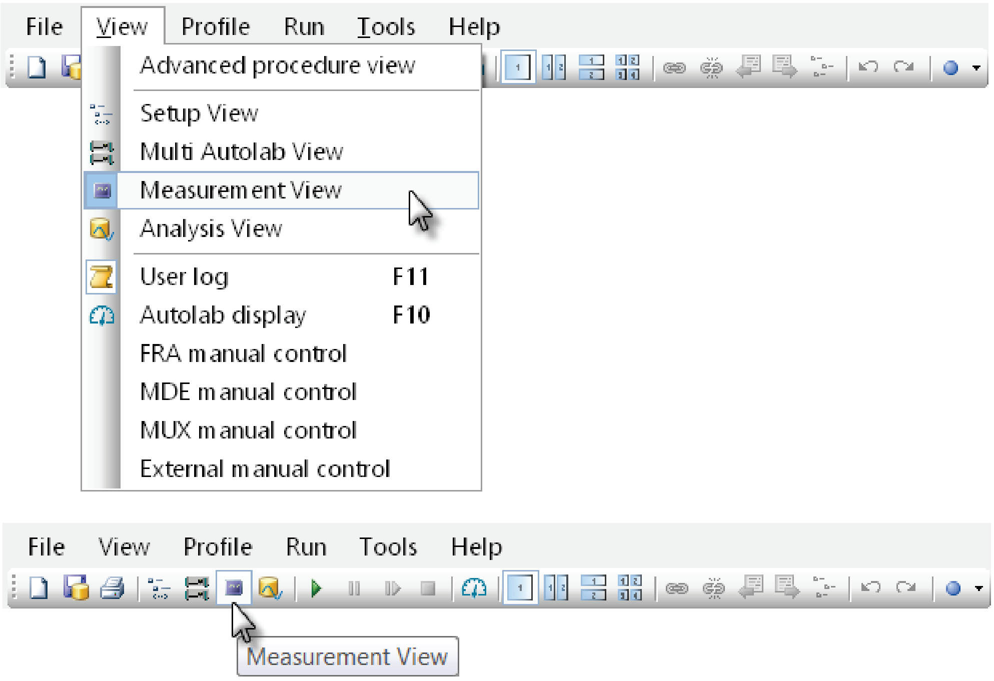


Figure 3.1 – Selecting the Measurement view

Real time information on the measured data and the procedure is shown and updated as the data is collected. Figure 3.2 provides an overview of the Measurement view.

Note

By default, Nova switches to the Measurement view automatically when the Start button is pressed. Refer to Section 1.6.4.3 for more information.

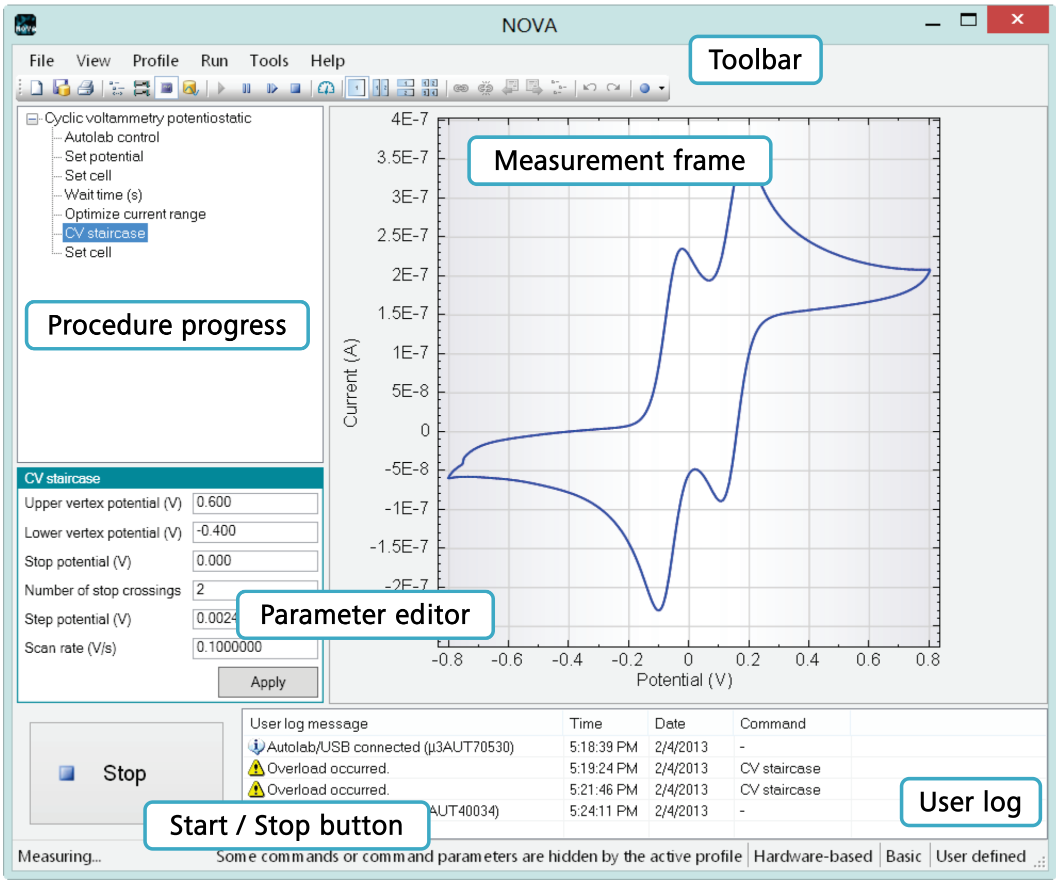


Figure 3.2 – An overview of the Measurement view

There are several highlighted areas in this view:

- **Measurement frame:** this is where the measured data points are plotted, in real time. The last measured point plotted in this frame is located by the cross marker (see the top right corner of the frame in Figure 3.2). A total of four plots are available in the Measurement frame. Dedicated toolbar buttons can be used to switch the Measurement frame to different plot arrangements (see Figure 3.3).

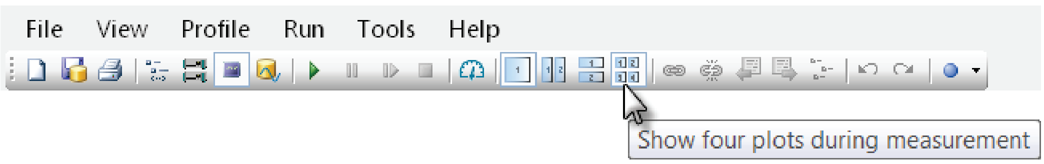


Figure 3.3 – Up to four different plots can be displayed in the measurement frame

Note

The contents of the plots are defined in the procedure setup (see next Section).

- **The procedure progress:** this frame displays a condensed version of the procedure that is currently running. Only the names of the commands are displayed. During the procedure, the progress frame can be updated, displaying information collected during the measurement. This frame is therefore helpful when running long procedures or procedures involving a number of repetitions.
- **The modifiable parameters:** this frame displays a list of modifiable parameters for the running command as well as the current values used for these parameters. Using this frame, it is possible to change one or more of these parameters while the command is being executed.

Note

Only the parameters of the running command can be modified in real time. Only specific parameters can be modified in real time. For a complete list of commands and parameters please refer to the Command list document, available from the Help menu.

The other items of the measurement view are the same as the other views (User log, start/stop button, toolbar).

3.1 – The procedure progress frame

During the measurement, an additional frame is provided in the measurement view. This frame, the procedure queue frame, displays a condensed version of the procedure. It shows the commands while the parameters are hidden from the view. The command of the procedure that is currently executed is highlighted.

As an example, the procedure progress of the standard Autolab linear polarization is shown in Figure 3.4.

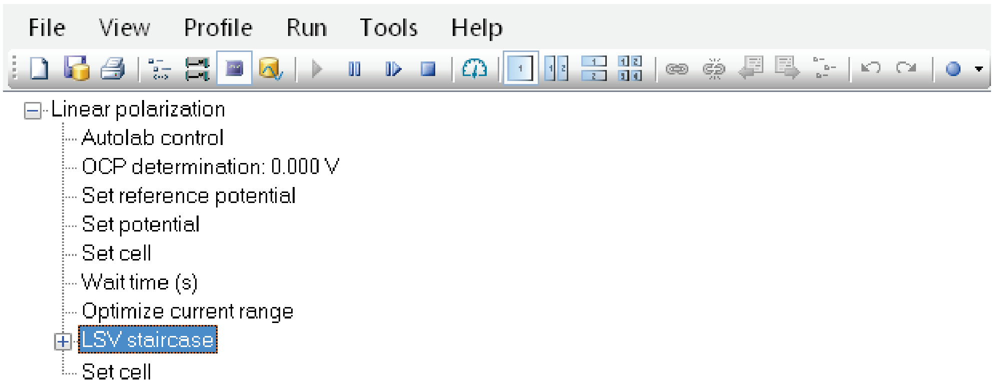


Figure 3.4 – The procedure progress frame during the standard Autolab linear polarization procedure (the *LSV staircase* command is highlighted, indicating that NOVA is currently running that command)

Note

The 0.000 V value displayed next the OCP determination command in the progress frame is updated once the value has been determined experimentally during the measurement.

Note

Working with views offers the possibility of performing other tasks while a measurement is running. The Autolab view is conveniently placed in an independent window and is always available to provide an overview of the measured data points and to provide the user with an interactive manual control of the instrument.

3.1.1 – Modification of command parameters in real time

The measurement progress frame displays a series of editable command parameters for the running command, if applicable. The displayed command parameters can be modified while the command is running. The parameters that can be modified are displayed in the dedicated frame (see Figure 3.5).

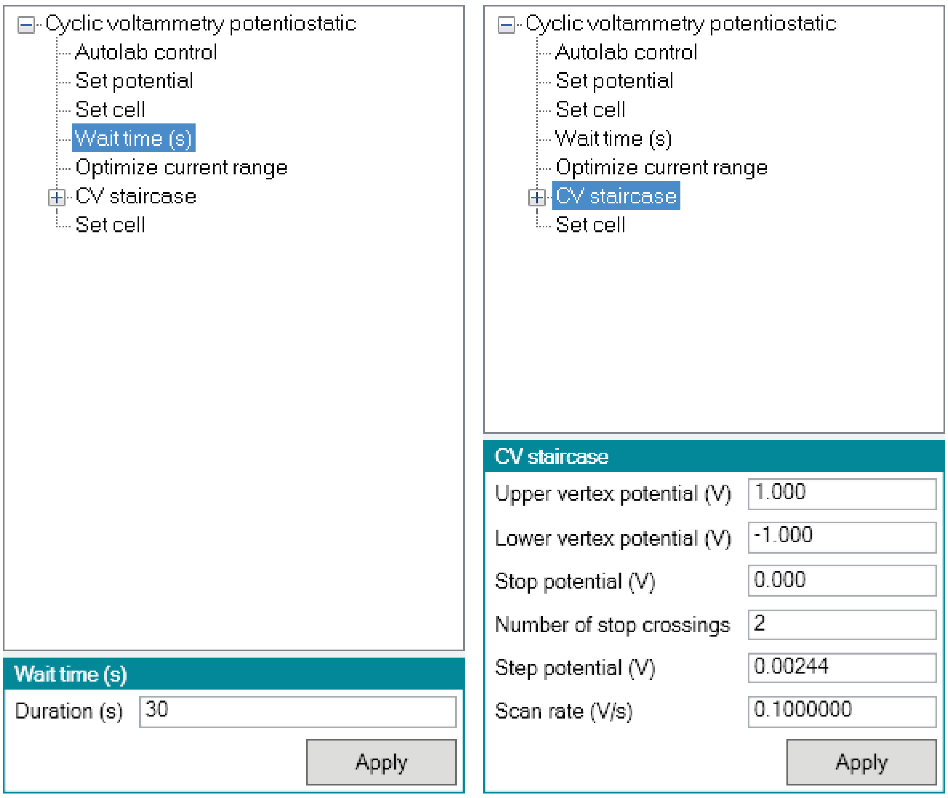


Figure 3.5 – Overview of the command parameters that can be changed during a measurement (left: *Wait time (s)* command, right: *CV staircase* command)

To modify a parameter, change the value of the parameter and press the 

button (see Figure 3.6).

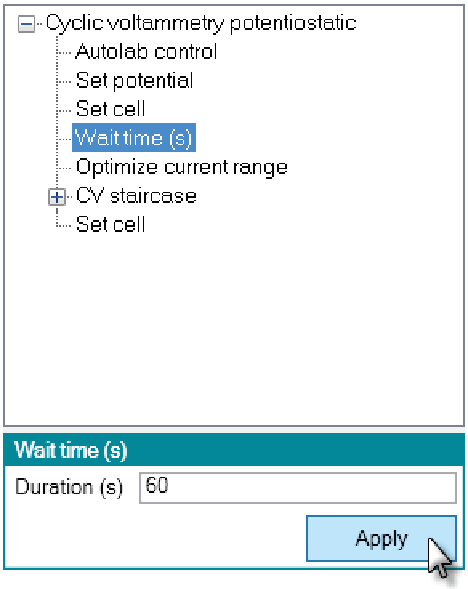


Figure 3.6 – Parameters can be changed by typing the new value and clicking the 

button

Note

Not all command parameters can be changed in real time. For more information on the parameters that can be modified in the measurement view, please consult the Command list document, available from the Help menu in Nova.

3.2 – Measurement plots

The measurement view is used to display the recorded data points during the experiment, or the results of calculation or data handling instructions added to the procedure. Up to four different plots can be used in the measurement view and it is possible to display one, two or four plots at the same time. The initial contents of these plots are defined in the procedure setup. Each Plot command added to the procedure in the Setup view has two parameters that define the plotting settings in the Measurement view (see Figure 3.7).

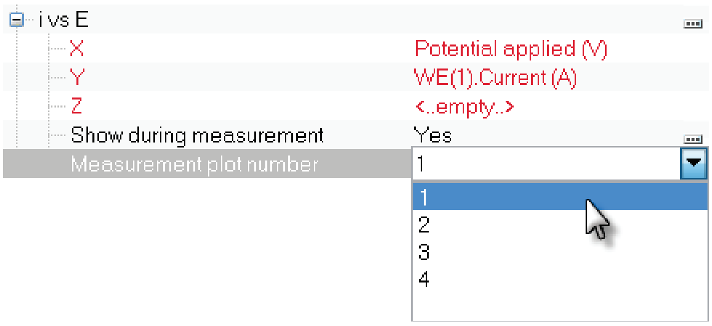


Figure 3.7 - The i vs E plot command used in the Autolab cyclic voltammetry potentiostatic procedure

The following parameters can be specified in the Setup view for each plot:

- **Show during measurement (Yes/No):** defines if the plot should be displayed in the measurement view during the experiment. When this parameter is set to No, then the plot is not shown during the measurement. The plot is, however, added to the data set and is available for plotting in the Analysis view.
- **Measurement plot number (1, 2, 3 or 4):** defines the location of the plot in the Measurement view. Up to four plots can be shown in the measurement view (see Figure 3.8).

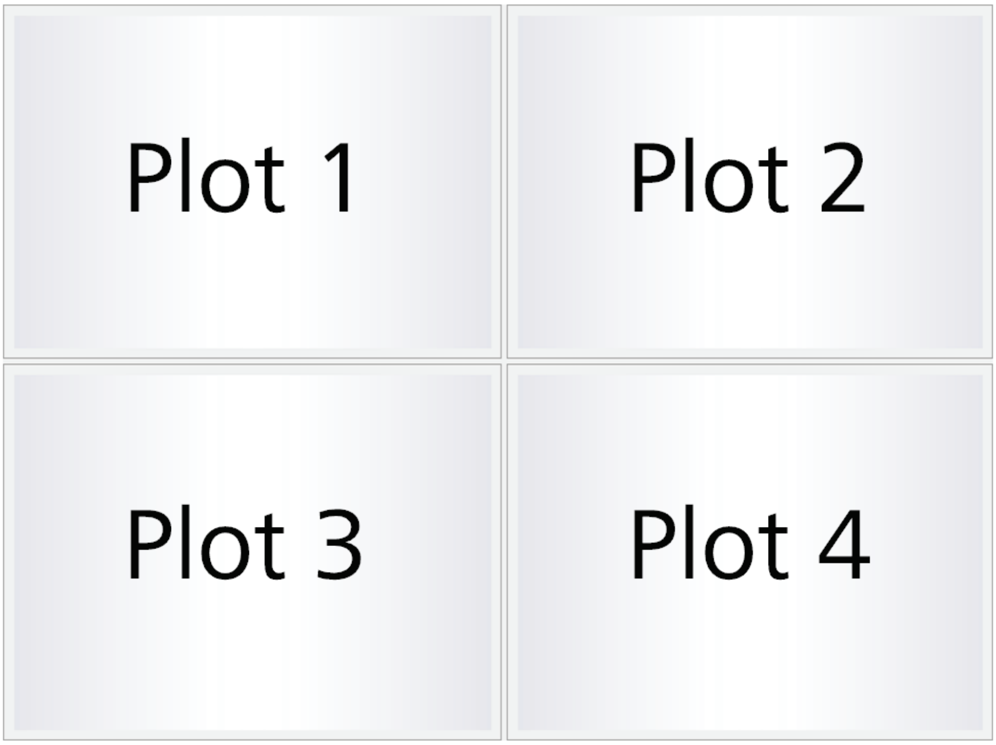


Figure 3.8 – The location of the four plots available in the Measurement view

Note

Click the , or button in the toolbar to display more than one plot in the measurement view.

Figure 3.9 shows an example of four plots displayed in the measurement view during an impedance spectroscopy measurement. Plot #1 corresponds to the Nyquist plot, plot #2 is the Bode plot (both modulus and phase), plot #3 is the Lissajous plot and plot #4 shows the resolution for both channels plotted versus time.

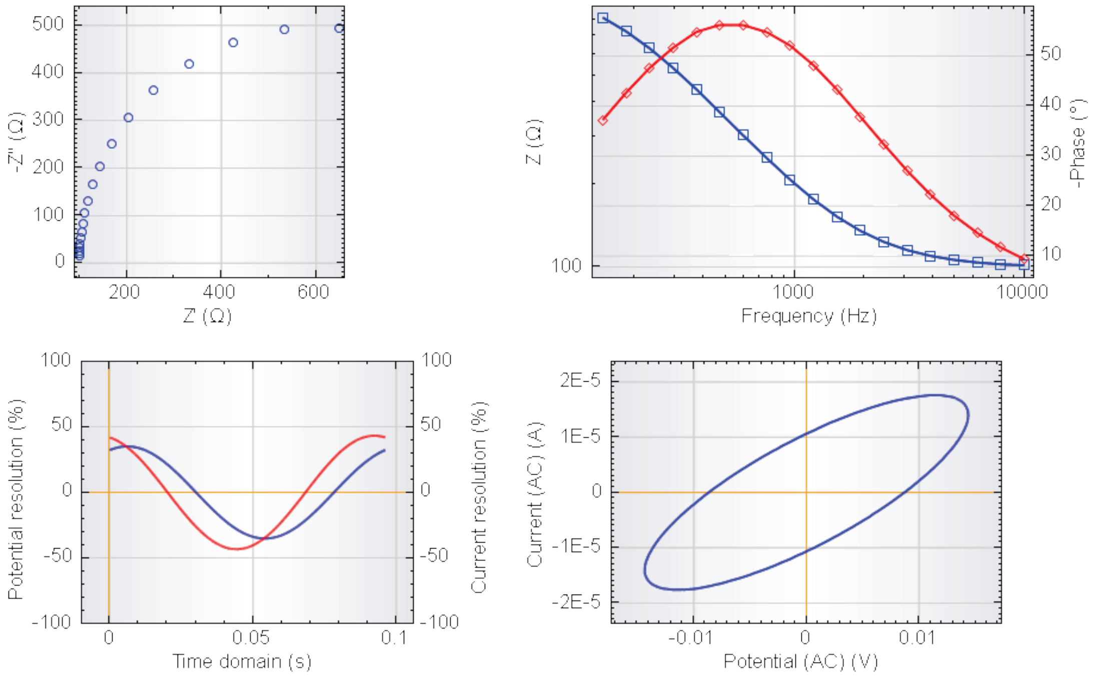


Figure 3.9 – Example of four plots shown in the measurement view during an impedance spectroscopy measurement

Note

The contents of each plot are removed whenever the type of signal used on the X-axis changes during a measurement.

3.2.1 – Adding and removing plots in real time

While the measurement is running, it is possible to add or remove plots from the measurement view. To add a plot, right-click a measurement command in the procedure progress frame and use the context-sensitive menu to add a new plot to the measurement, as shown in Figure 3.10.

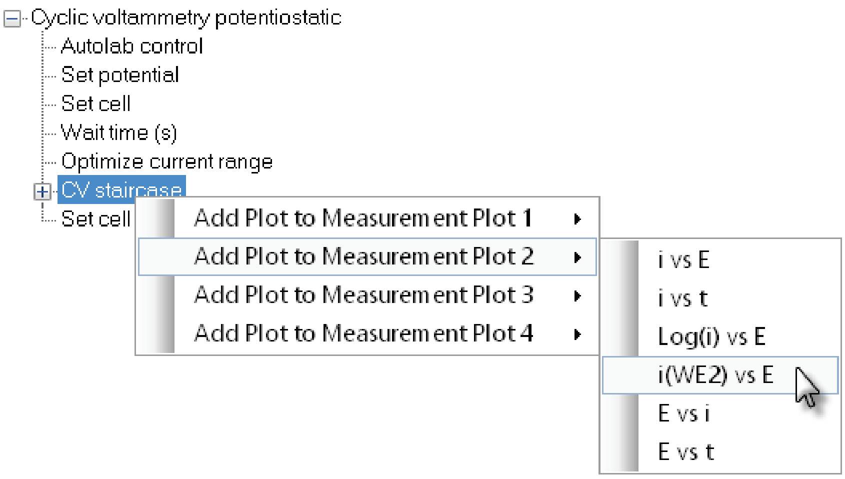


Figure 3.10 – The right-click menu can be used to add a new plot to the measurement

The list of available plots shown in the context menu depends on the signals sampled during the measurement. In Figure 3.10, the WE(2).Current, recorded through the Bipotentiostat module, is present in the signal sampler for the *CV staircase* command. It is therefore possible to add the i(WE2) vs E plot to the measurement, on plot #2.

To remove a plot, right-click on the plots shown below the command and select the Delete option from the context menu (see Figure 3.11).

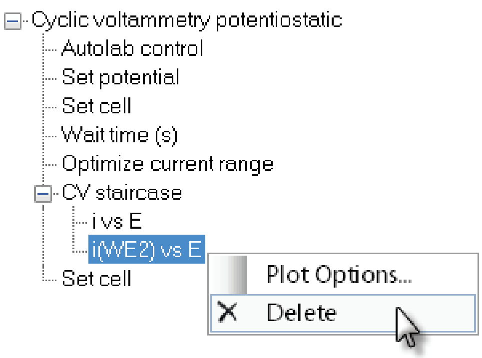


Figure 3.11 – Deleting a plot from the measurement view

This will remove the plot from the measurement view.

Note

The data is saved at the end of the measurement using the plots existing at the end of the experiment. If plots are removed during the experiment, these will not be saved. If plots are added, these will be saved.

3.2.2 – Modification of plot options in real time

It is possible to modify the plot options during the measurement. Right-clicking a plot in the measurement view displays a context menu (see Figure 3.12). Select the Plot Options to change the settings of the selected plot.

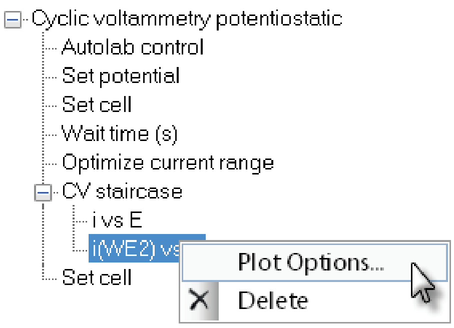


Figure 3.12 – Modifying the plot options during a measurement

Note

The data is saved at the end of the measurement using the plots existing at the end of the experiment. If plot options are changed during the experiment, the last used plot options will be saved.

3.3 – Clearing the measurement view and zooming

During the measurement, the clear measurement plot button can be clicked at any time to clear all the plots in the measurement view. The plotting of the data points will resume after the plot has been cleared. The measured data points are however kept in memory.

Note

The clear plot button can be extended by clicking the arrow button located next to the clear plot button. This will show a context menu with which any one of the four plots can be cleared (see Figure 3.13).

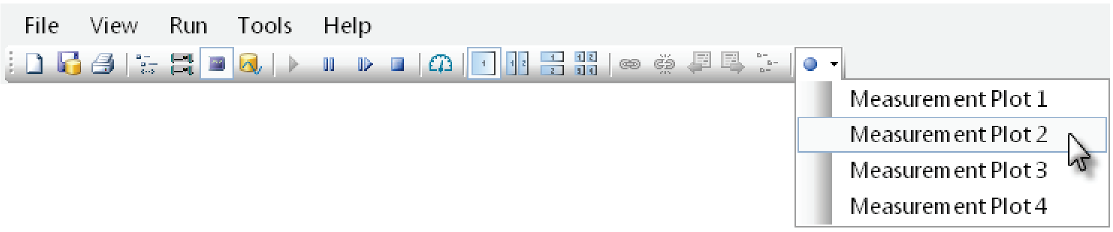


Figure 3.13 – It is possible to clear a specific plot in the measurement view by extending the Clear plot button

It is also possible to zoom in and out in the measurement view. By default, NOVA always tries to automatically adjust the X and Y scaling in order to plot all the data points. However, it is sometimes convenient to be able to zoom in on a particular zone of the plot in order to observe fine details of the curve.

It is only possible to zoom in and out on the data if the Enable zooming / moving option is enabled. To enable this option, right-click the plot area and select the Enable Zooming / Moving option from the menu (see Figure 3.14).

Zooming in on the data can be done by dragging a box around the zone of interested or by scrolling the wheel mouse[26]. Changing the scaling during the measurement disables the automatic rescaling. To resume this function, press the F4 key on the keyboard.

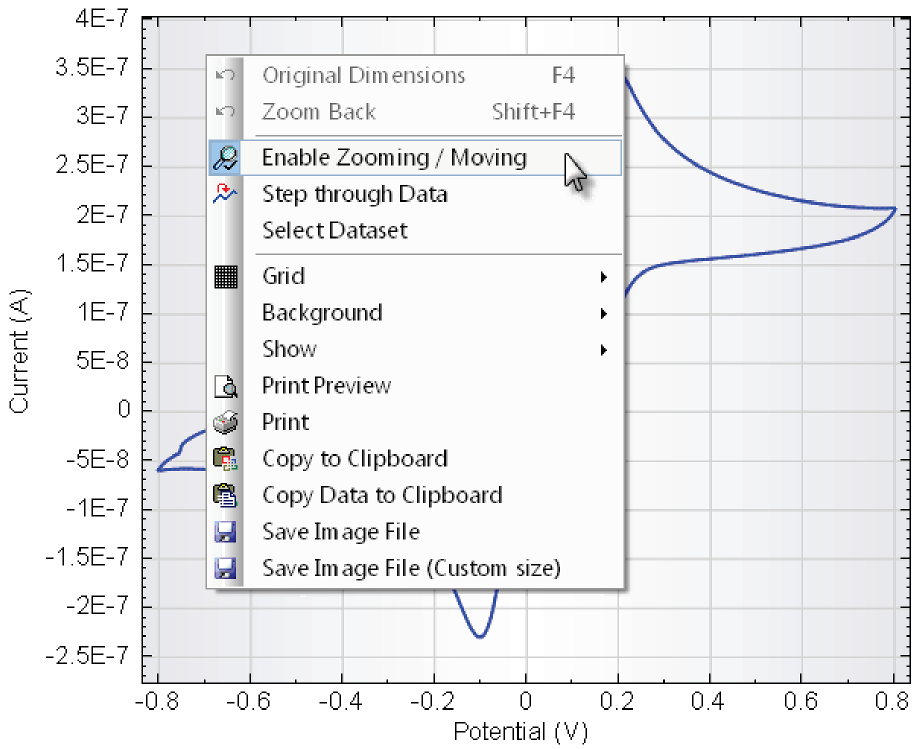


Figure 3.14 – Right click the plot to select the Enable Zooming / Moving option

Note

Additional options are available in the context menus in the measurement view. More information is provided in Chapter 4 (Section 4.7.3 to 4.7.13).

[26] If a wheel mouse is not available, it is possible to use the + and – keys on the keyboard to achieve the same results.

3.4 – The Autolab display

The Autolab display[27] is an extra window which can be displayed in each of the four views (Setup, Multi Autolab, Measurement and Analysis). It has two purposes: the first is the manual control of the instrument and the second function is the display of real time information about measured data during an experiment.

The Autolab display can be opened by selecting the Autolab display option from the View menu, by using the F10 key or by clicking the dedicated button in the toolbar (see Figure 3.15).

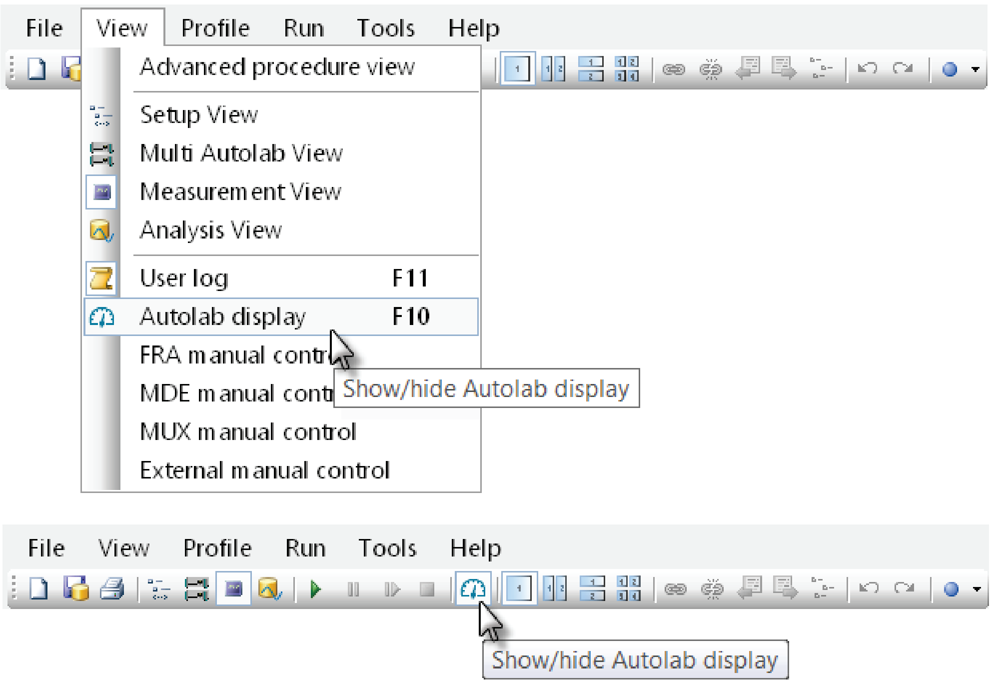


Figure 3.15 – Choose the Autolab display option from the view menu or use the dedicated button in the toolbar to open the Autolab display window

3.4.1 – Manual control

The manual control of the Autolab is built into the lower part of the Autolab display. A series of labels are displayed in this section of the view (see Figure 3.16). These labels can be clicked to change the settings of the instrument.

[27] The colors used in the Autolab display can be set using the Tools – Options menu.

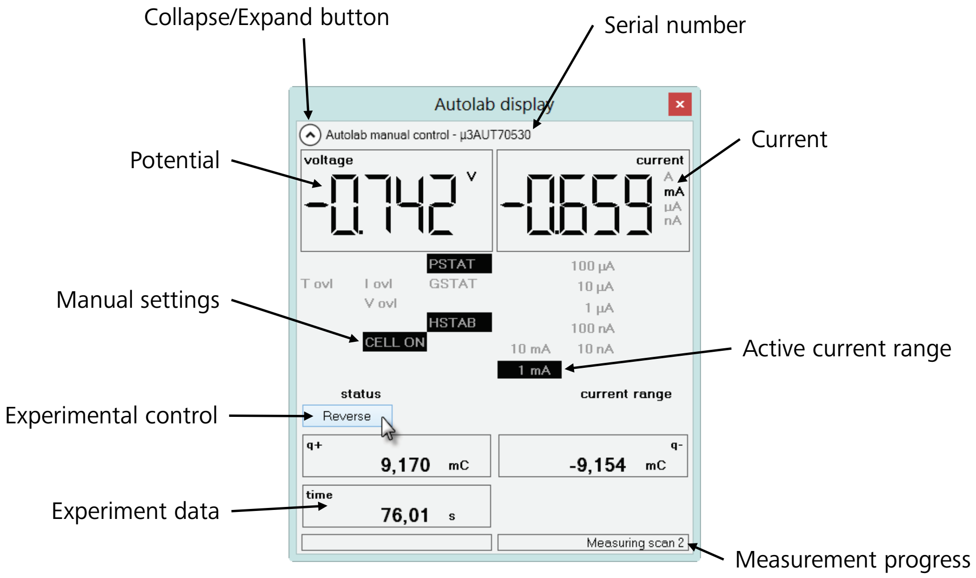


Figure 3.16 – An overview of the Autolab display during a CV measurement (µAutolab III)

The available current ranges are displayed on the right-hand side of the Autolab display. The highest and lowest possible current range depend on the hardware settings (in the case of Figure 3.16, the instrument is a µAutolab III, therefore the highest current range is 10 mA). The serial number of the instrument is located in the header of the Autolab display window[28].

The selected current range is highlighted. The Reverse button located in the Autolab display can be used to reverse the scan direction[29].

The active settings are highlighted. It the case of Figure 3.16, the settings are:

- Potentiostat mode (PSTAT label)
- Current range 1 mA (1 mA label)
- High stability on (HSTAB label)
- Cell on (CELL ON label)

Most of the labels of the manual control are *interactive*. A setting of the Autolab potentiostat can be changed by clicking the corresponding label.

Clicking the labels of the manual control will also update the settings displayed on the front panel of the instrument.

The information displayed at the bottom of the Autolab display window depends on the experimental method.

[28] This is the indentifying serial number of the instrument. When the Autolab is used in combination with an external USB interface, the serial number of the USB interface is shown.
[29] This option is only available for cyclic voltammetry and linear sweep voltammetry staircase.

3.4.2 – Additional control panels

Additional control panels can be added to the Autolab display window, by selecting the corresponding option from the view menu. For example, if a FRA2 or FRA32M module is available, the FRA manual control option can be selected to display the manual control of this module (see Figure 3.17).

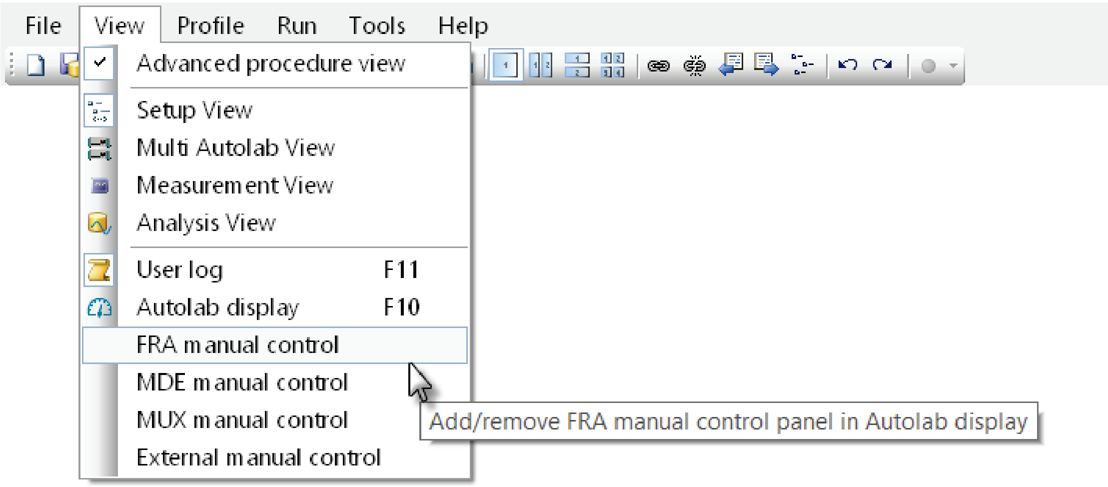


Figure 3.17 – Adding the FRA manual control to the Autolab display

The FRA manual control panel can be used to control the FRA2 or FRA32M module and to perform impedance measurements (see Figure 3.18).

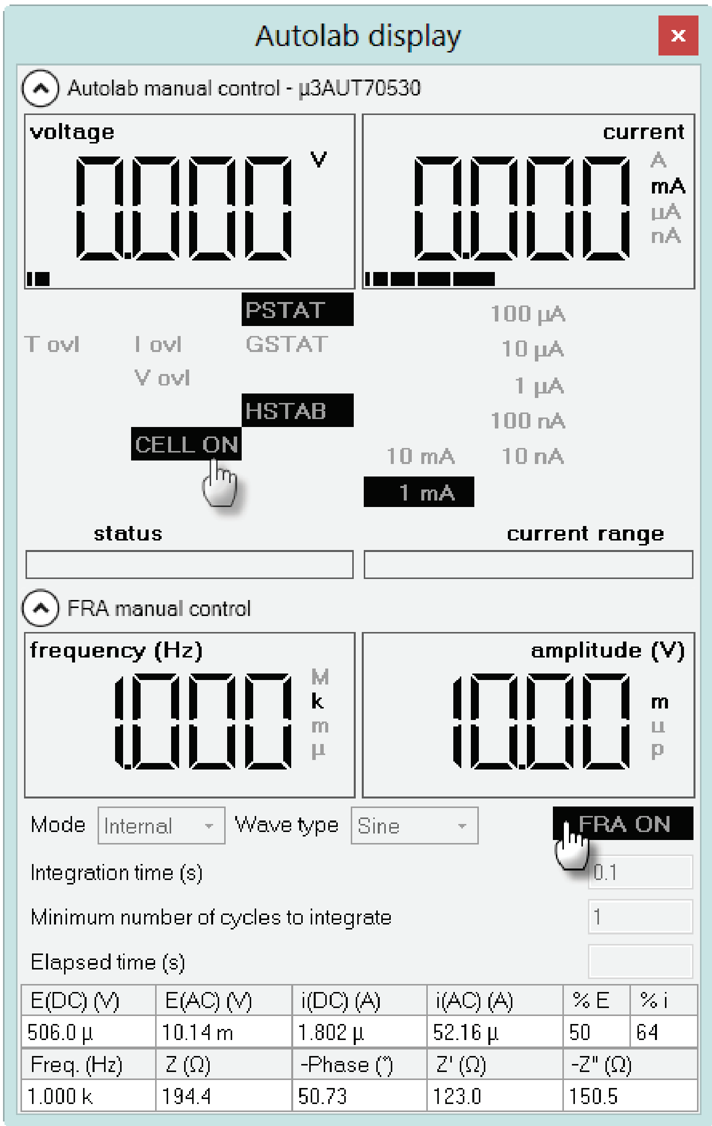


Figure 3.18 – The FRA manual control panel can be used to control the FRA2 module through the Autolab display

This panel also displays measured impedance values during a frequency scan[30].

3.4.3 – Collapsible panels

At any time, it is possible to click on the button located in the top left corner of an Autolab display panel to collapse the contents of this panel in order to hide the information (see Figure 3.19).

[30] More information can be found in the Impedance spectroscopy tutorial, available from the Help – Tutorials menu.

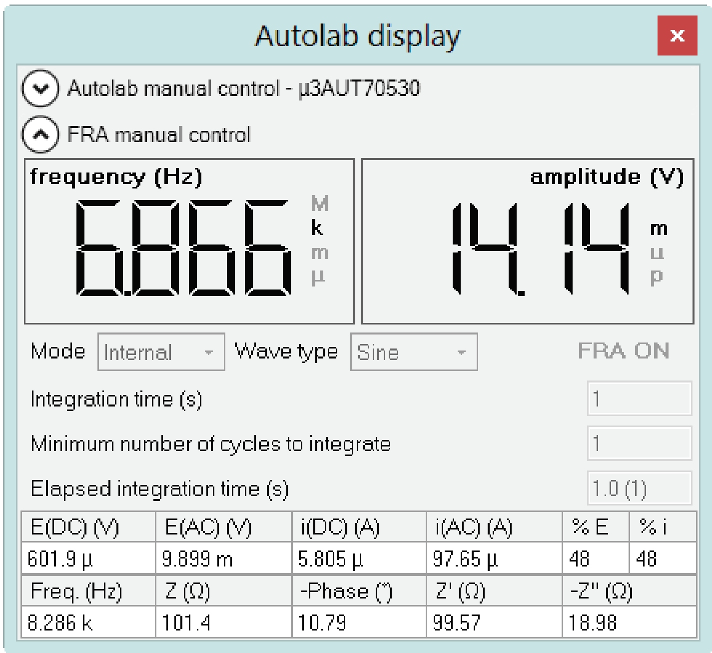


Figure 3.19 – Collapsing the Autolab control panel

3.4.4 – Information during measurements

During a measurement, the Autolab view displays values of several relevant electrochemical signals. These values are displayed at the bottom of the display. The displayed information depends on the type of measurement. Figure 3.19 shows the additional information displayed during a FRA measurement, while Figure 3.16 displays information about the anodic and cathodic charge during a cyclic voltammetry measurement.

3.4.5 – Information while not measuring

When no measurements are performed, the Autolab display shows the real time values of the measured current and potential, as well as the noise levels associated with both signals. The noise is represented by the noise gauges, present below the numerical values of current and potential (see Figure 3.20).

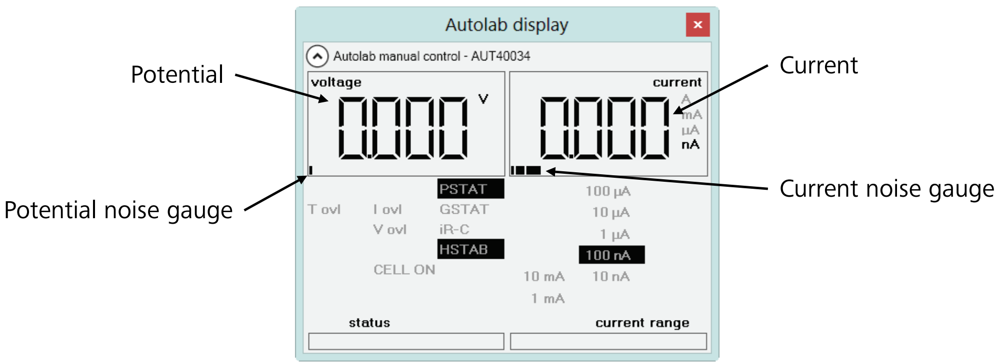


Figure 3.20 – The noise gauges are indicating the noise levels qualitatively

The noise gauges provide real time information concerning the quality of the signals reported in the Autolab display. The noise is represented by a horizontal bar graph. It represents the standard deviation of the signals measured during one full measurement cycle (20 ms for systems working at 50 Hz and 16.67 ms for systems working at 60 Hz). The noisier a signal, the larger the standard deviation will be and the noisier the signal will be.

4 – Analysis view

Data analysis is performed in the **Analysis view**. The Analysis view can be accessed at any time, even during an ongoing measurement, by clicking the corresponding button in the toolbar or selecting the Analysis view from the View menu (see Figure 4.1).

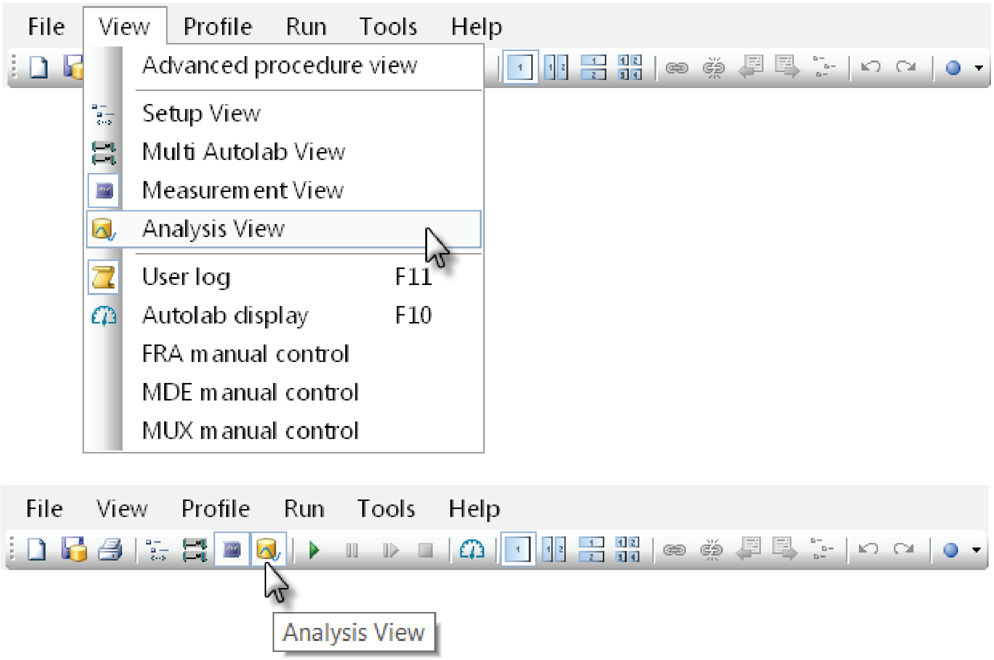


Figure 4.1 – Switching to the Analysis view

This view, like the other views of the NOVA software, has a specific layout, with several areas of interest. An overview of the analysis view is given in Figure 4.2.

Note

Like all the frames in Nova, the frames in the data analysis view can be resized. Moreover, the user log can be toggled off using the view menu. This allows you to maximize the size of the Data analysis frame for a better overview of the data.

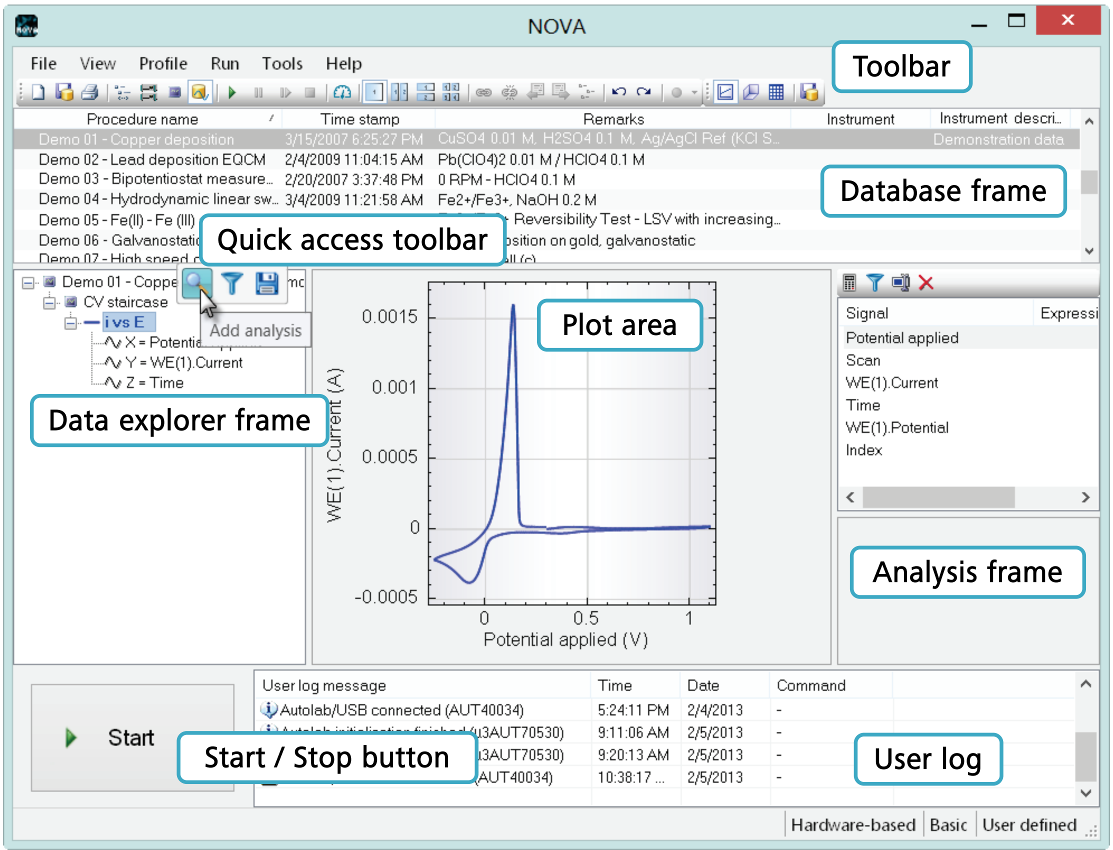


Figure 4.2 – Overview of the Analysis View

The Start/Stop button, toolbar and the User Log are both common to all views of NOVA. Other noteworthy areas of the Analysis View are:

- **Database:** displays the list of available data sets that can be used in the data analysis view. Each entry of the database has a name, a time stamp and a remarks field.
- **Data explorer:** displays the data set(s) currently selected for data analysis.
- **Plot area:** displays the selected data points in a 2D or 3D plot.
- **Analysis frame:** displays the control parameters and the results of data analysis tools.

4.1 – Introduction

This chapter of the user manual explores the Analysis view and provides in-depth information about this essential part of the software. Some examples are provided in this chapter and the corresponding data sets are available in the ***Demo database*** folder, which is created during the installation of NOVA 1.10. This database contains all the examples used in this chapter.

4.1.1 – The database

To use the database, click the button on the right of the area displaying the path of the Measured data database. Change the path to the Demo Database located in the ***Program Files\Metrohm Autolab\NOVA 1.10\Shared DataBases*** folder (see Figure 4.3). Click the OK button to confirm the change in the path of the database and click the OK button in the database management window to close it.

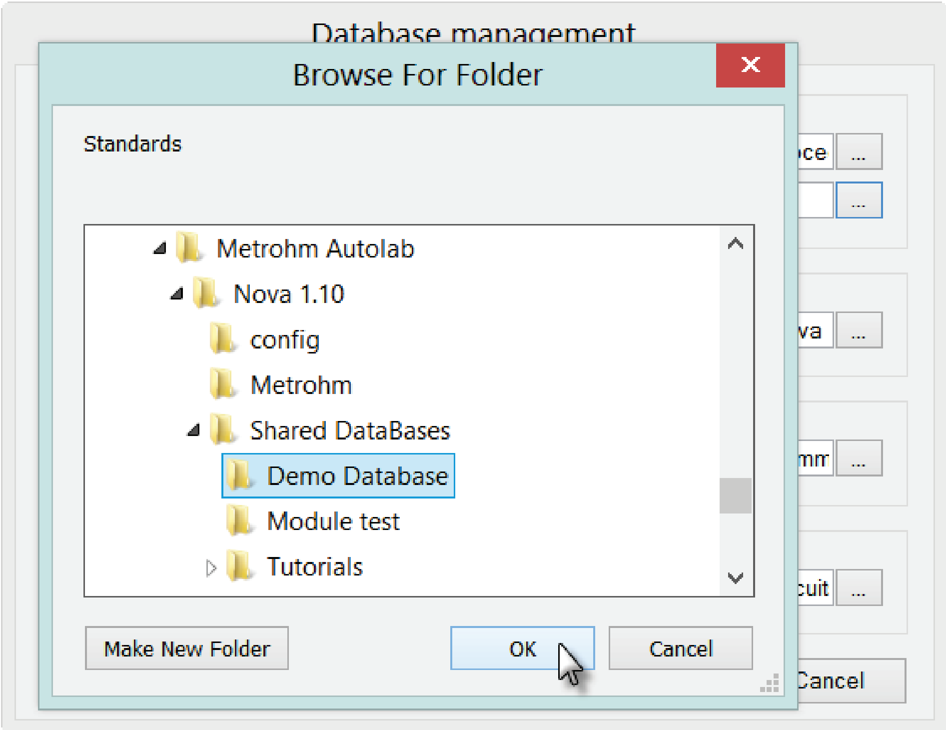


Figure 4.3 – Loading the Demo database by setting as the Data database

The demonstration database has been set as a *Data* database. Its contents are displayed in the database frame of the Analysis view (see Figure 4.4).

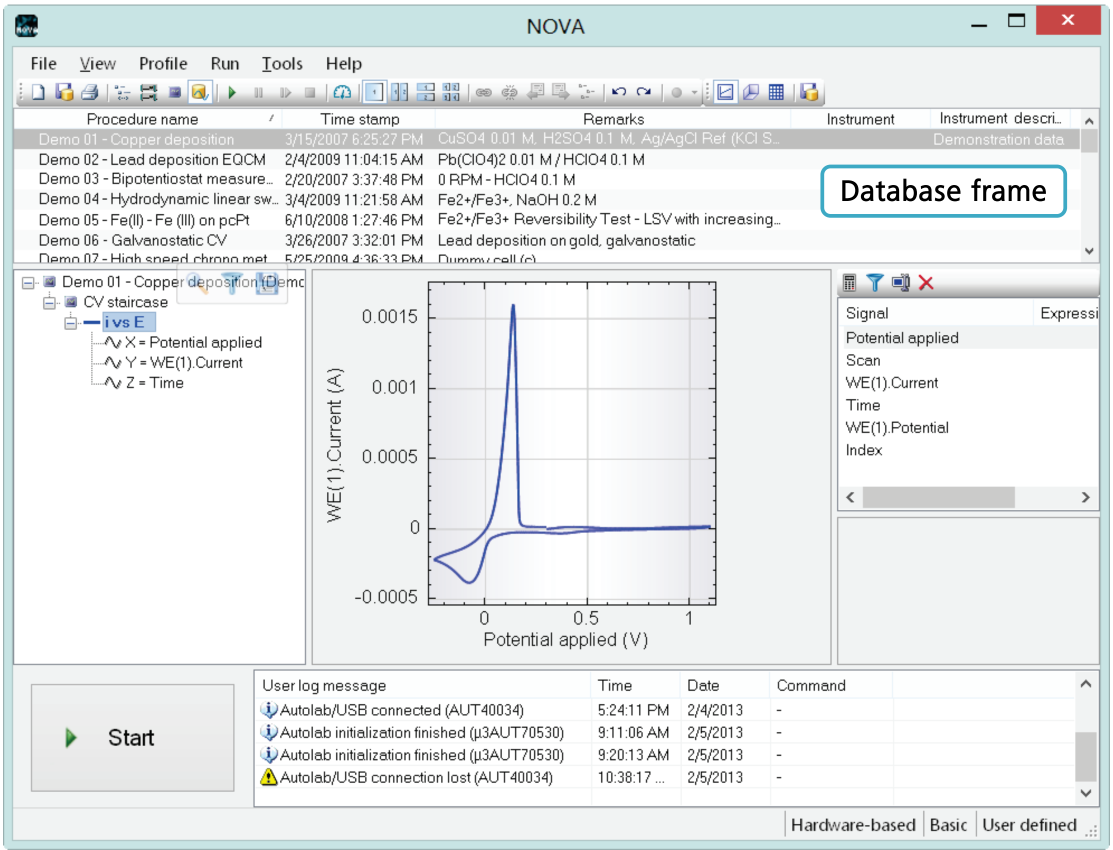


Figure 4.4 – The contents of the database are displayed in the database frame in the analysis view

The demonstration database contains 20 data sets, logged by Procedure Name, Time stamp, Remarks, Instrument serial number[31] and Instrument description. The displayed date corresponds to the time stamp of the experiment. The data sets can be sorted, ascending or descending, using one of the five columns.

Figure 4.5 shows a detailed picture of the database.

[31] This is the serial number used by the software to identify the instrument. For measurements performed on an Autolab with an external USB interface, the serial number of the USB interface will be used to identify the device.

Procedure name	Time stamp	Remarks	Instrument	Instrument description
Demo 01 - Copper deposition	3/15/2007 6:25:27 PM	CuSO4 0.01 M, H2SO4 0.1 M, Ag/AgCl R…	AUT83072	Demonstration data
Demo 02 - Lead deposition EQCM	2/4/2009 11:04:15 AM	Pb(ClO4)2 0.01 M / HClO4 0.1 M	AUT83072	Demonstration data
Demo 03 - Bipotentiostat measurement - Pt/Pt Rin…	2/20/2007 3:37:48 PM	0 RPM - HClO4 0.1 M	AUT83072	Demonstration data
Demo 04 - Hydrodynamic linear sweep voltammetr…	3/4/2009 11:21:58 AM	Fe2+/Fe3+, NaOH 0.2 M	AUT83072	Demonstration data
Demo 05 - Fe(II) - Fe (III) on pcPt	6/10/2008 1:27:46 PM	Fe2+/Fe3+ Reversibility Test - LSV with in…	AUT83072	Demonstration data
Demo 06 - Galvanostatic CV	3/26/2007 3:32:01 PM	Lead deposition on gold, galvanostatic	AUT83072	Demonstration data
Demo 07 - High speed chrono methods (Fast ADC)	5/25/2009 4:36:33 PM	Dummy cell (c)	AUT83072	Demonstration data
Demo 08 - Chrono methods (ADC164) - 20 steps	2/20/2007 8:40:08 AM	Factory standard procedure.	AUT83072	Demonstration data
Demo 09 - Chrono methods (ADC164) with variabl…	2/20/2007 9:37:06 AM	Combination of steps and levels	AUT83072	Demonstration data
Demo 10 - Differential pulse measurement	5/25/2009 12:14:00…	Example for baseline correction, peak sea…	AUT83072	Demonstration data
Demo 11 - Hydrodynamic FRA with OCP determin…	4/8/2010 12:23:00 PM	With Autolab RDE at 1000 RPM	AUT83072	Demonstration data
Demo 12 - Imported pcPt GPES data	6/10/2008 7:50:38 PM	Polycrystalline platinum in HClO4 0.1 M		Imported demonstration data
Demo 13 - Imported pcPt GPES data	6/10/2008 7:53:08 PM	EtOH oxidation on polycrystalline Pt in HC…		Imported demonstration data
Demo 14 - FC-(CH2)2-FC in ACN/CH2Cl2	6/10/2008 8:09:37 PM	Ferrocene in Acetonitrile / Chloroform		Imported demonstration data
Demo 15 - UME LSV	6/10/2008 8:30:23 PM	Use the smooth function		Imported demonstration data
Demo 16 - FRA impedance	4/9/2010 4:54:02 PM	Dummy cell (c)	µ3AUT70530	Demonstration data
Demo 17 - Imported FRA data	6/10/2008 8:27:23 PM	frademo.dfr		Imported demonstration data
Demo 18 - Aniline electropolymerization	5/5/2009 11:57:13 AM	ESPR measurement, Au disk	µ3AUT70530	Demonstration data
Demo 19 - Cyclic voltammetry Fe2+/Fe3+	3/17/2009 5:35:06 PM	Cyclic voltammetry potentiostatic: no extr…	µ3AUT70530	Demonstration data
Demo 20 - Iron screw in seawater	4/5/2009 3:42:46 PM	Corrosion rate analysis demo data		Imported demonstration data

Figure 4.5 – Detailed view of the demonstration database

4.1.1.1 – Sorting data

The highlighted symbol located on the right-hand side of the Procedure Name column header (triangle pointing up symbol) indicates that the data sets are currently sorted by name, ascending. Clicking on the Procedure Name column header will switch the name sorting to descending (see Figure 4.6). The displayed symbol will change to triangle pointing down.

Procedure name	Time stamp	Remarks	Instrument	Instrument description
Demo 20 - Iron screw in seawater	4/5/2009 3:42:46 PM	Corrosion rate analysis demo data		Imported demonstration data
Demo 19 - Cyclic voltammetry Fe2+/Fe3+	3/17/2009 5:35:06 PM	Cyclic voltammetry potentiostatic: no extr…	µ3AUT70530	Demonstration data
Demo 18 - Aniline electropolymerization	5/5/2009 11:57:13 AM	ESPR measurement, Au disk	µ3AUT70530	Demonstration data
Demo 17 - Imported FRA data	6/10/2008 8:27:23 PM	frademo.dfr		Imported demonstration data
Demo 16 - FRA impedance	4/9/2010 4:54:02 PM	Dummy cell (c)	µ3AUT70530	Demonstration data
Demo 15 - UME LSV	6/10/2008 8:30:23 PM	Use the smooth function		Imported demonstration data
Demo 14 - FC-(CH2)2-FC in ACN/CH2Cl2	6/10/2008 8:09:37 PM	Ferrocene in Acetonitrile / Chloroform		Imported demonstration data
Demo 13 - Imported pcPt GPES data	6/10/2008 7:53:08 PM	EtOH oxidation on polycrystalline Pt in HC…		Imported demonstration data
Demo 12 - Imported pcPt GPES data	6/10/2008 7:50:38 PM	Polycrystalline platinum in HClO4 0.1 M		Imported demonstration data
Demo 11 - Hydrodynamic FRA with OCP determin…	4/8/2010 12:23:00 PM	With Autolab RDE at 1000 RPM	AUT83072	Demonstration data
Demo 10 - Differential pulse measurement	5/25/2009 12:14:00…	Example for baseline correction, peak sea…	AUT83072	Demonstration data
Demo 09 - Chrono methods (ADC164) with variabl…	2/20/2007 9:37:06 AM	Combination of steps and levels	AUT83072	Demonstration data
Demo 08 - Chrono methods (ADC164) - 20 steps	2/20/2007 8:40:08 AM	Factory standard procedure.	AUT83072	Demonstration data
Demo 07 - High speed chrono methods (Fast ADC)	5/25/2009 4:36:33 PM	Dummy cell (c)	AUT83072	Demonstration data
Demo 06 - Galvanostatic CV	3/26/2007 3:32:01 PM	Lead deposition on gold, galvanostatic	AUT83072	Demonstration data
Demo 05 - Fe(II) - Fe (III) on pcPt	6/10/2008 1:27:46 PM	Fe2+/Fe3+ Reversibility Test - LSV with in…	AUT83072	Demonstration data
Demo 04 - Hydrodynamic linear sweep voltammetr…	3/4/2009 11:21:58 AM	Fe2+/Fe3+, NaOH 0.2 M	AUT83072	Demonstration data
Demo 03 - Bipotentiostat measurement - Pt/Pt Rin…	2/20/2007 3:37:48 PM	0 RPM - HClO4 0.1 M	AUT83072	Demonstration data
Demo 02 - Lead deposition EQCM	2/4/2009 11:04:15 AM	Pb(ClO4)2 0.01 M / HClO4 0.1 M	AUT83072	Demonstration data
Demo 01 - Copper deposition	3/15/2007 6:25:27 PM	CuSO4 0.01 M, H2SO4 0.1 M, Ag/AgCl R…	AUT83072	Demonstration data

Figure 4.6 – Sorting the data sets by name, descending

The data can be sorted on Procedure name, Time stanp, Remarks, Instrument serial number and Instrument description.

4.1.1.2 – Data filtering

The database also provides a filtering option that can be useful when exploring a database with a lot of entries. By using the filter option, it is possible, for example, to select all measurements that have been obtained using a specific instrument.

Click the *Demo 01 – Copper deposition* entry of the database and right-click on the **Instrument** header. Select **Filter with selected dropped on row**, using a **Like** option (see Figure 4.7).

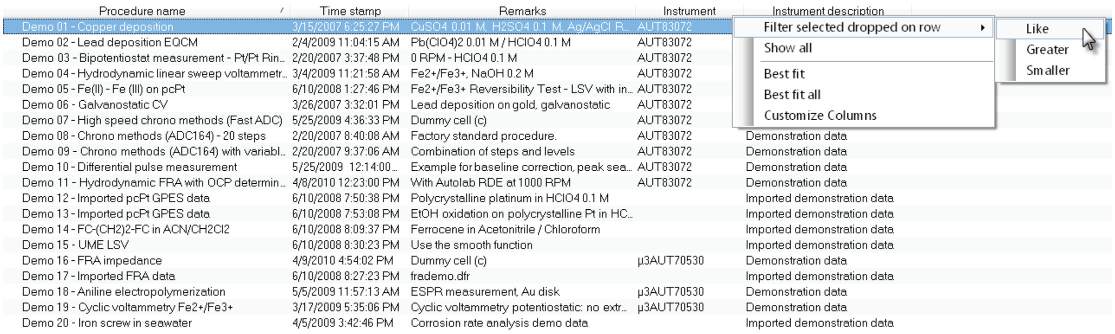


Figure 4.7 – Filtering the database using the Instrument column

The contents of the database will be updated, displaying only the database entries obtained with the instrument with serial number AUT83072 (see Figure 4.8).

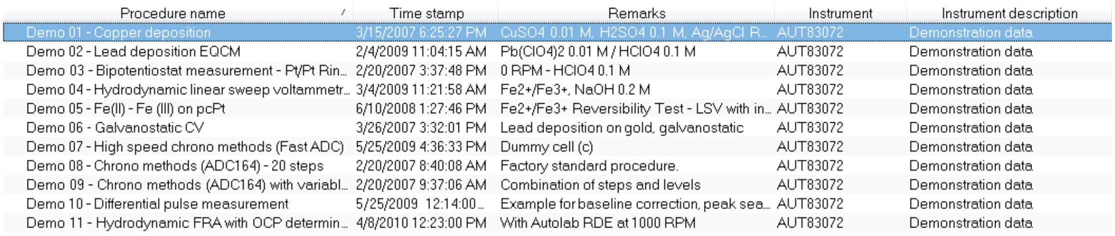


Figure 4.8 – The filtered demonstration database

The eleven data sets displayed in the filtered database have being obtained with the same instrument as the one used for the *Demo 01 – Copper deposition* entry of the database (AUT83072)). By using the filtering option described above, only the data sets for which the serial numbers are the same, are displayed.

To remove the filter, right-click the database header and select the **Show all** option from the context menu (see Figure 4.9). This will restore the database to its original content, displaying all the data sets.

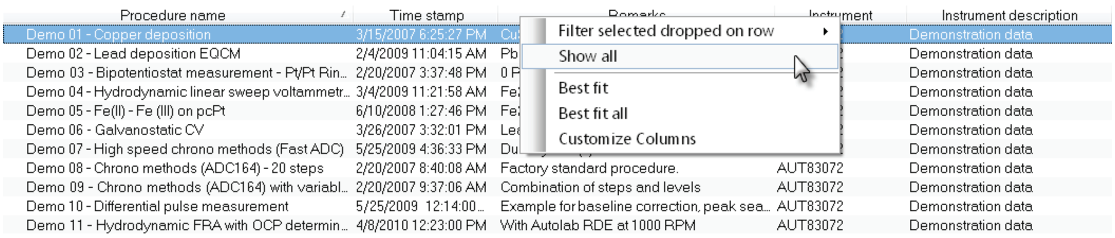


Figure 4.9 – Select the Show all option to view the entire contents of the database

Note

Use the Best fit and Best fit all to automatically adjust the width of the columns in the database.

4.1.1.3 – The data repository

An additional feature of the database storage system is the **data repository**. With the repository, it is possible to create one or more internal backups of a database entry. This makes it possible to recover the original data and it can also be used as audit trail. To store data in the repository, right-click the corresponding entry in the database and choose the *Store in Repository* option from the context menu (see Figure 4.10).

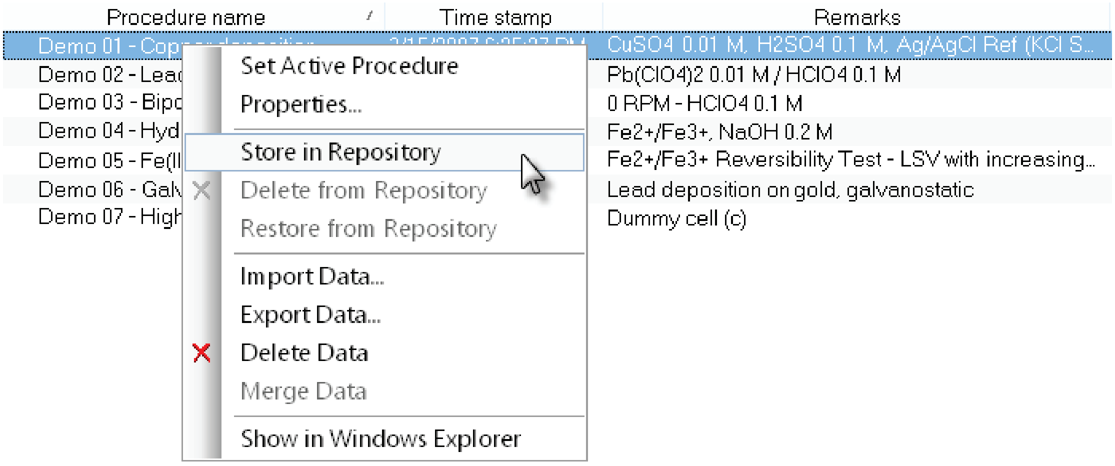


Figure 4.10 – Select the Store in Repository option to create a backup of the database entry

The store in repository option adds a copy of the original data set in the database using the backup creation time as the time stamp (see Figure 4.11).

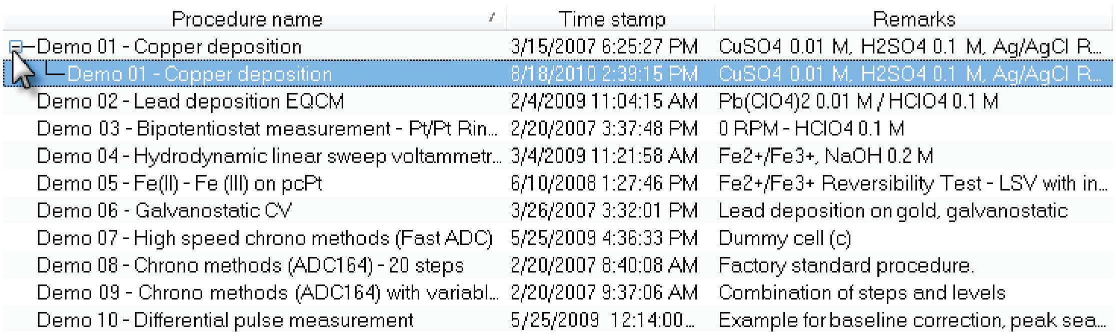


Figure 4.11 – The repository backup creates a copy of the original data set

Once a backup has been added to the repository, it is possible to modify the original data set and restore it at any time by choosing the *Restore from Repository* (see Figure 4.12). It is also possible to create more than one backup in the data repository.

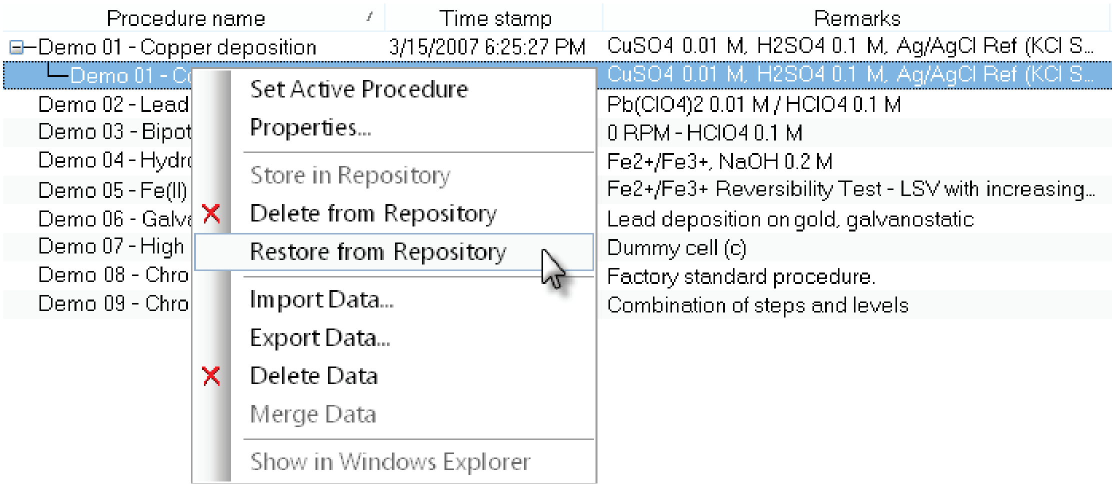


Figure 4.12 – Restoring the original data set from the repository backup

Note

Using the Restore from repository removes any modification of the current data set and restores the data set to the backed up status.

4.1.1.4 – Merging data

The database also allows the merging of two or more files. When database entries are merged, a new file containing the procedures and the data from the merged files will be copied to the new file. This can be used to involve the data from two or more different measurements in a calculation or other data handling steps described in this chapter.

To merge two or more data files in the database, select the files by clicking them while holding the CTRL key pressed. The selected files will be highlighted in the database frame (see Figure 4.13).

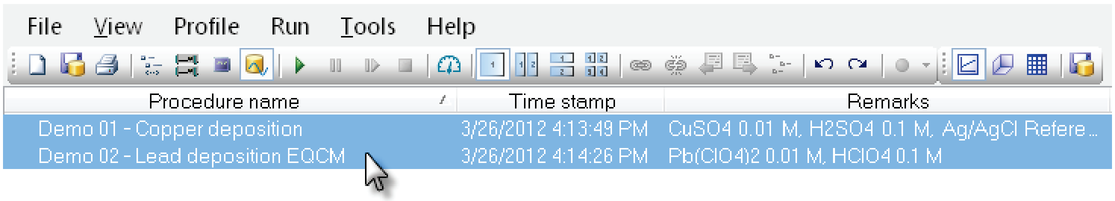


Figure 4.13 – Selecting multiple database entries

Right-click the selected database entries and select the Merge data option from the context menu (see Figure 4.14).

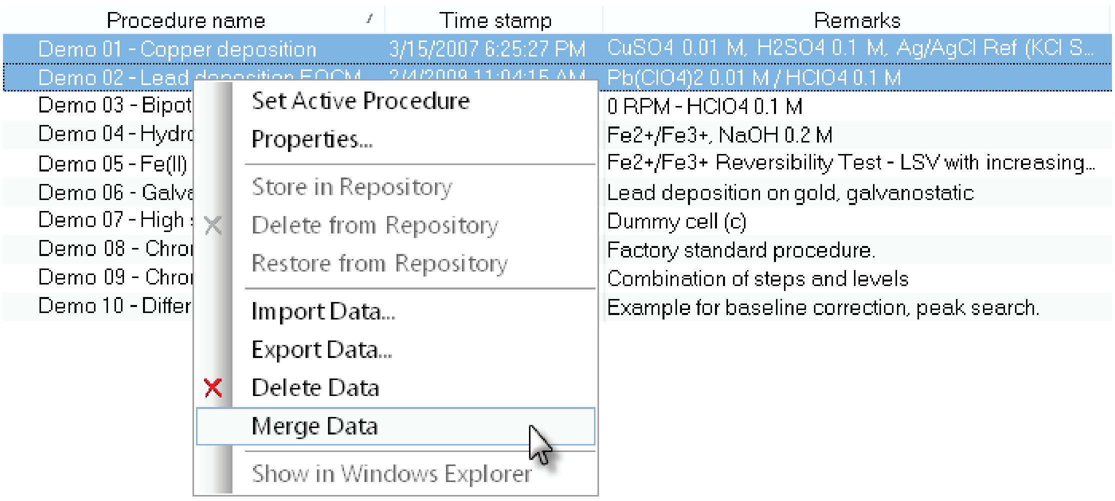


Figure 4.14 – Right-click the selected entries and select the Merge Data option

A popup window will be displayed (see Figure 4.15).

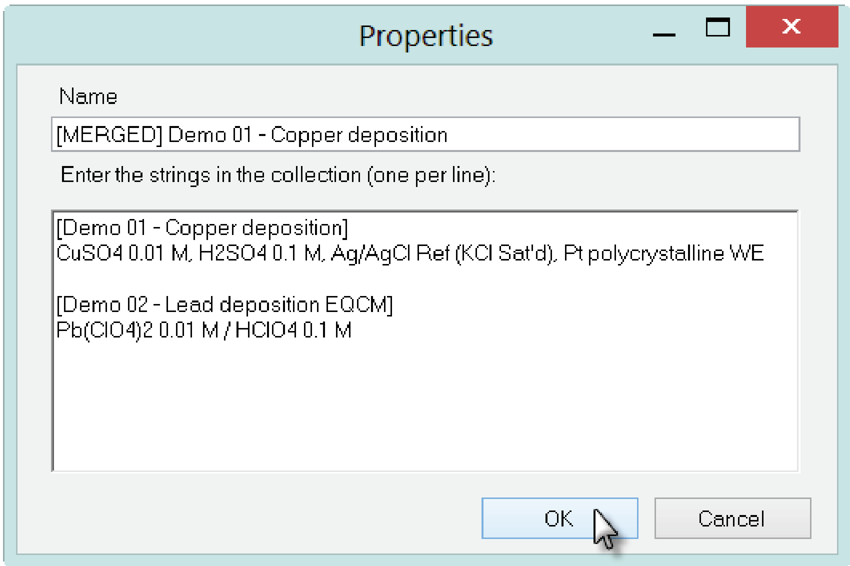
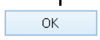


Figure 4.15 – A popup dialog is displayed when the files are merged

Using the window, a new database filename can be specified and the remarks field for the merged file can be edited. Pressing the  button and closes the editor, adding a file to the database (see Figure 4.16).

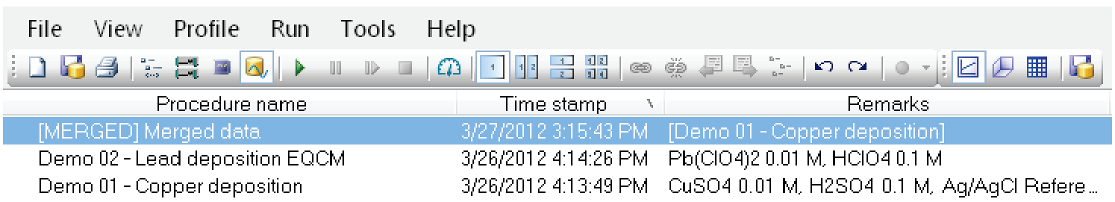


Figure 4.16 - The merged data is added to the dabadase

The new file will contain the data from the source data files.

Note

Some Windows versions do not allow saving of data in the *C:\Program Files* folder.

4.1.1.5 – Import and Export data

It is also possible to Import additional files into the database or to Export data files from the database as single files (with a *.nox* extension). Importing and exporting of data files is done through the right-click menu.

To export a data file, right-click the corresponding entry in the database and select the Export data option from the context menu (see Figure 4.17).

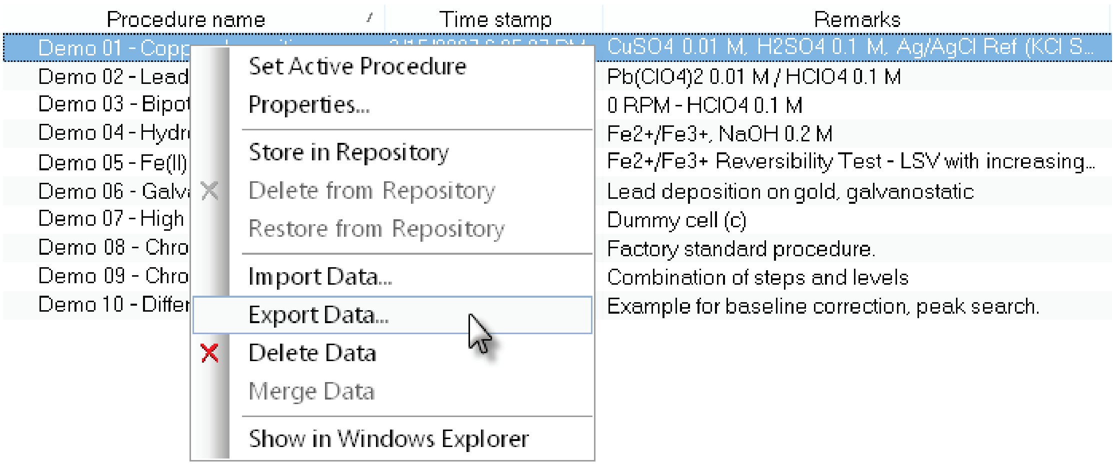


Figure 4.17 – Exporting a data file

A name and a location for the file can be specified (see Figure 4.18).

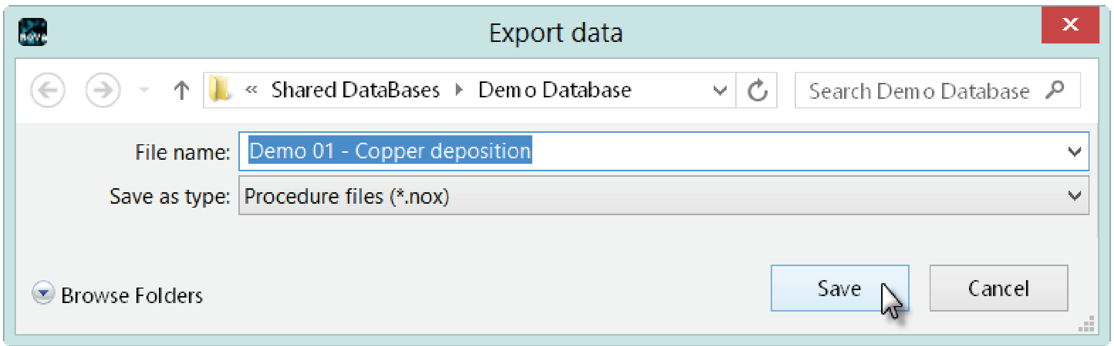


Figure 4.18 – Specifying a name and location for the exported file

Note

It is possible to export multiple files at the same time, although an individual name and location must be specified for each file.

To import a data file, right-click anywhere in the database frame and select the Import data option from the context menu (see Figure 4.19).

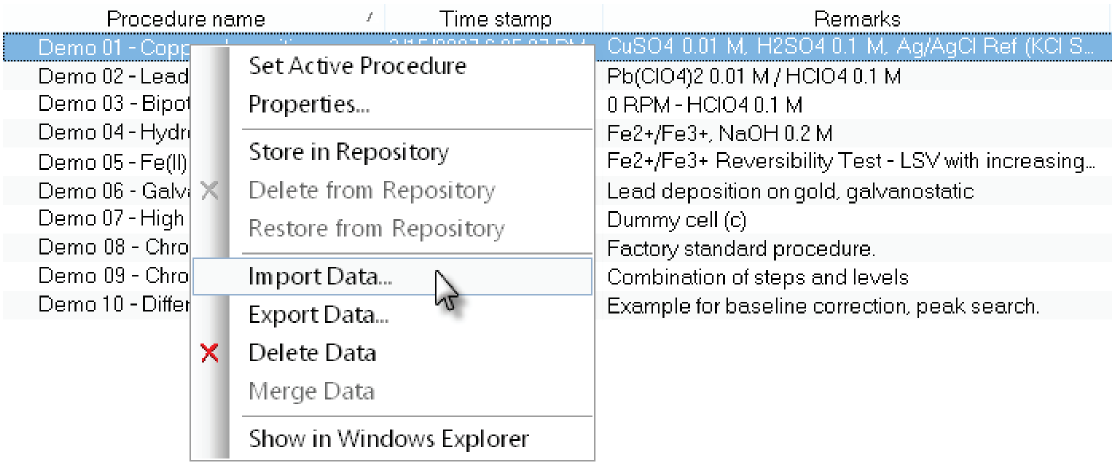


Figure 4.19 – Right-click anywhere to import a data file

To import a data file, right-click anywhere in the database frame and select the Import data option from the context menu (see Figure 4.20).

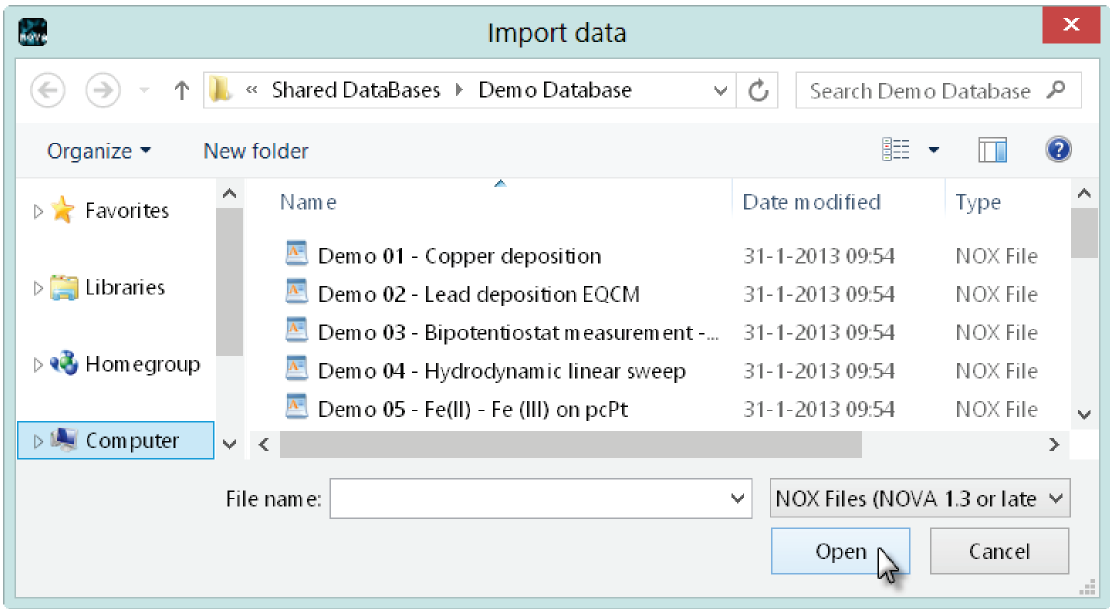


Figure 4.20 – The dialog shown during the importing of data files

Note

It is possible to import multiple files at the same time, although an individual name and location must be specified for each file.

Warning

NOVA 1.10 is compatible with data measured with previous versions of NOVA. Data and procedures from previous versions can be directly imported in the current version of NOVA. A conversion tool, which can be used to convert data from the previous versions to the 1.10 format, is available. Refer to the **Upgrading files from previous version** tutorial, available from the Help menu, for more information.

4.1.1.6 – Location of files

The location of a single file of the database can be quickly determined by right-clicking the entry in the database frame and selecting the *Show in Windows explorer* option from the context menu (see Figure 4.21).

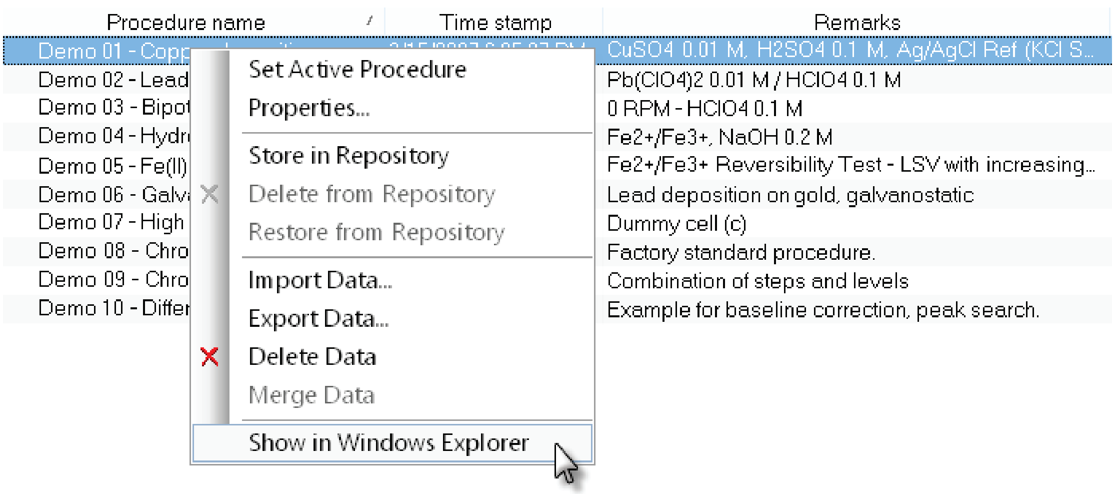


Figure 4.21 – The *Show in Windows explorer* option provides a shortcut to the location of a database entry

A Windows explorer window will open, showing the location of the selected file (see Figure 4.22).

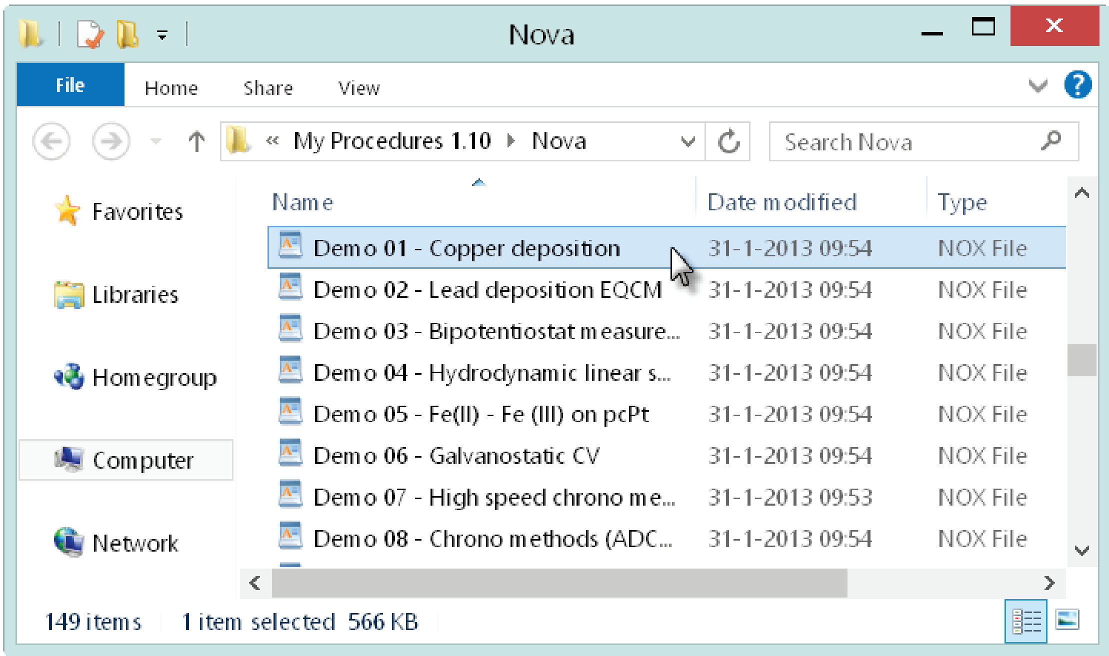


Figure 4.22 – The file is shown in Windows explorer

4.1.1.7 – Deleting files

To delete a data file from the database, right-click the corresponding database entry and select the Delete Data option from the context menu (see Figure 4.23).

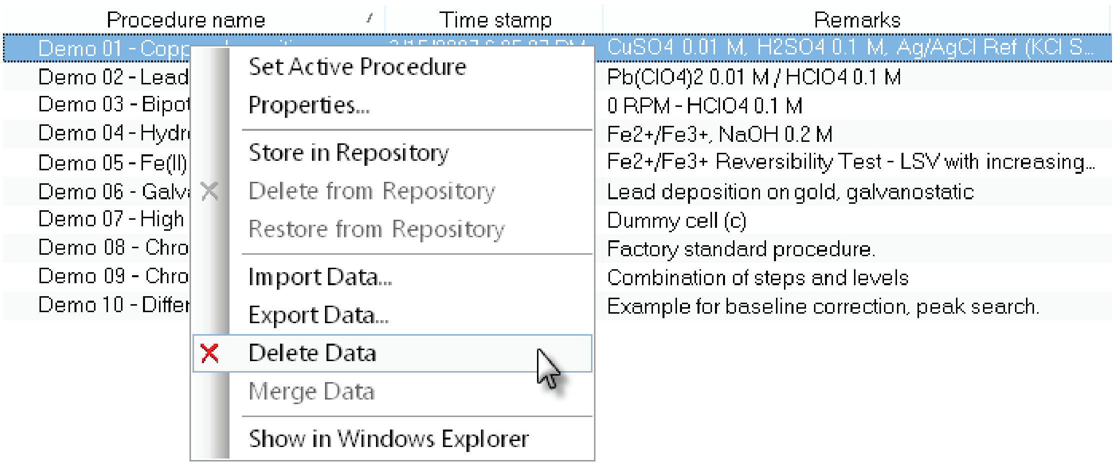


Figure 4.23 – Deleting data from the database

Note

It is possible to delete multiple files at the same time. Deleted data files are automatically moved to the Recycle bin.

4.2 – Plotting the data in 2D

The basic display of measured data sets in NOVA is a 2D plot. The way the data is rendered in the 2D plot depends on the available signals. Assuming that a given data set consists of three different signals (Current, Potential and Time), the data set can be displayed in six different 2D plots:

- Current vs. Potential or Potential vs. Current
- Current vs. Time or Time vs. Current
- Potential vs. Time or Time vs. Potential

The data analysis view also includes a **data grid**, which is a spreadsheet-like advanced calculator that includes an expression builder that can be used to construct new signals, based on existing signals. This means that the plotting possibilities of NOVA are almost infinite (the use of the data grid will be detailed in section 4.9 of this chapter).

4.2.1 – Loading and viewing data sets from the database

The first step required to view the measured data from a given data set of the database is to **load** this data set in the Data explorer frame. To do this, double click the corresponding entry of the database. This will copy the data set in the explorer frame (see Figure 4.24).

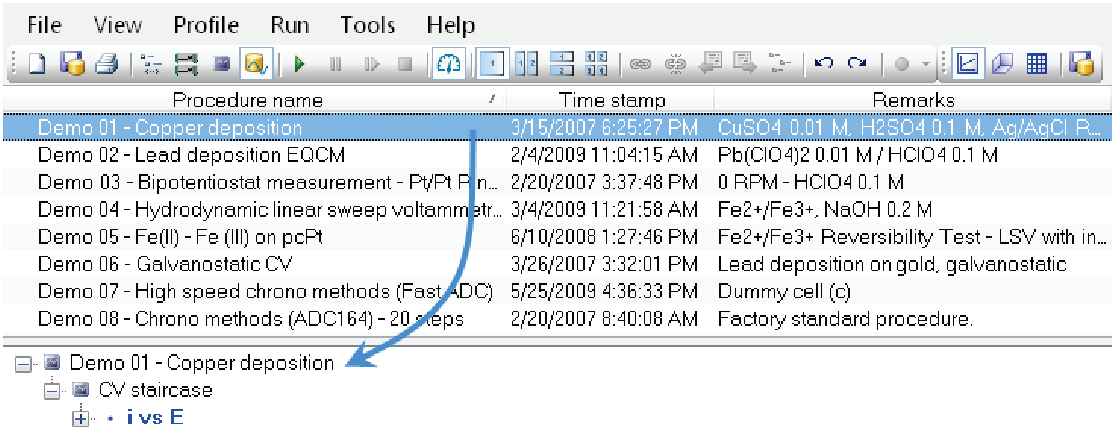


Figure 4.24 – Loading a data set from the database into the data explorer

Note

The *Demo 01 – Copper deposition* entry of the demo database contains a cyclic voltammogram for copper deposition on platinum, recorded in 0.1 M $CuSO_4$ in 0.1 M H_2SO_4 solution. The reference electrode was a Ag/AgCl (KCl sat'd) and the counter electrode was a platinum wire.

Once a data set has been added to the data explorer frame, it can be plotted in a 2D plot. By default, a cyclic voltammogram experiment is displayed by plotting the applied potential on the X-axis and the measured current on the Y-axis. To view the measured data using this setting, click the blue i vs E line. This will plot the experimental data from the data set as a blue **Point plot**, shown in Figure 4.25.

Note

Nova assigns default axis labels based on the signals used to plot the measured data.

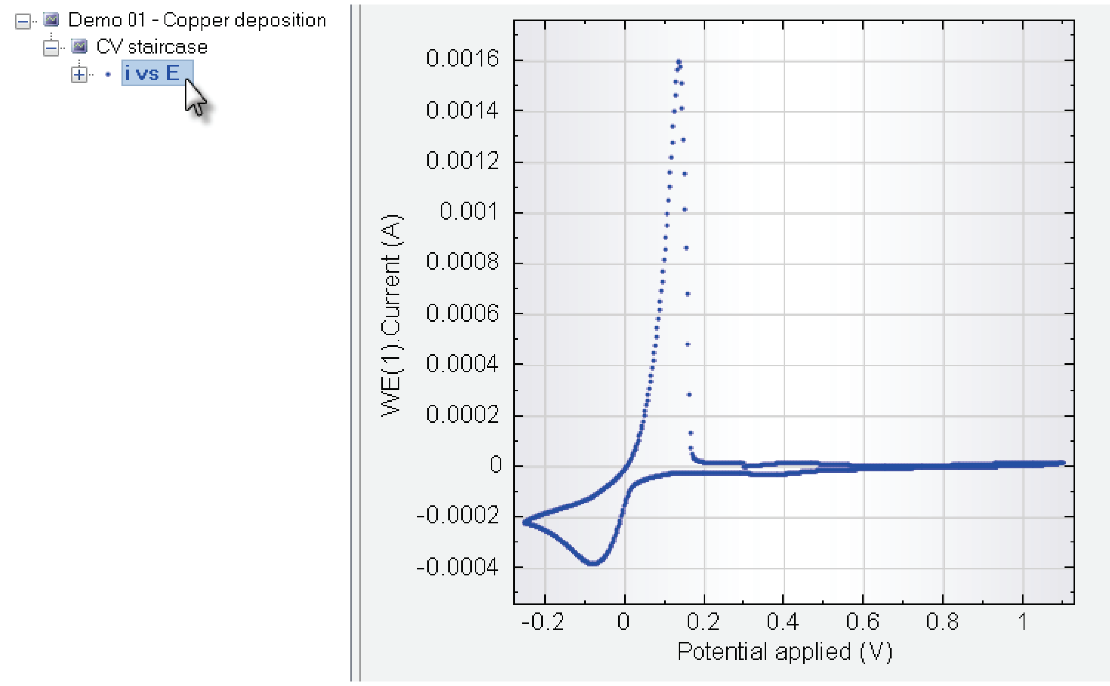


Figure 4.25 – Plotting example #1 in a 2D plot

Note

Clicking the i vs E line will change it to bold lettering, and it will be highlighted.

- The bold lettering indicates that the data set is currently **plotted** in the data analysis frame.
- The highlighted status indicates that the data set plotted in the data analysis frame is currently also the **active plot** (in the case of an overlay of several plots, only one can be active).

4.2.2 – Changing the data set display settings

The way the data is displayed is controlled by the signals used for the plot. The data from example #1, shown in Figure 4.25, is displayed using the Potential applied signal for the X-axis, the WE(1).Current signal for the Y-axis and the Time signal for the Z-axis, although the latter in not shown in the 2D plot. To change these display settings, expand the signals list by clicking the ⊞ symbol, right-click on any of the signal and select the desired signal from the list. In the example shown in Figure 4.26, the plot settings are changed in order to display the potential applied as a function of time.

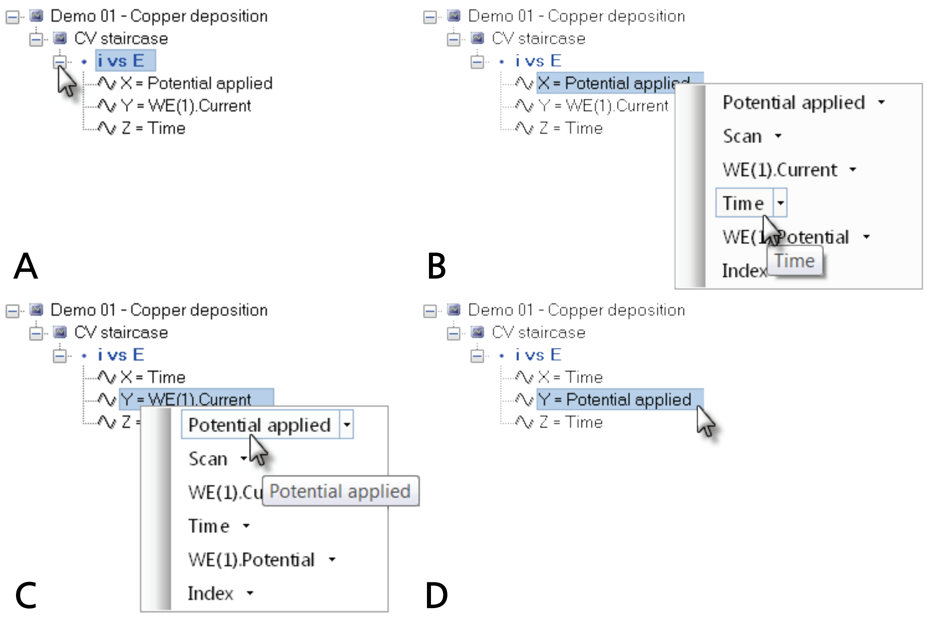


Figure 4.26 – Changing the display settings to WE(1).Potential applied (Y-axis) vs. Time (X-axis). A – Expanding the signals list; B – Changing the signal displayed on the X-axis; C – Changing the signal displayed on the Y-axis; D – The new 2D plot for the data from example #1

Changing the display settings in order to plot the potential in function of time as described in the picture above will change the display of the data set and a familiar saw-tooth potential profile of a cyclic voltammogram will be plotted (see Figure 4.27).

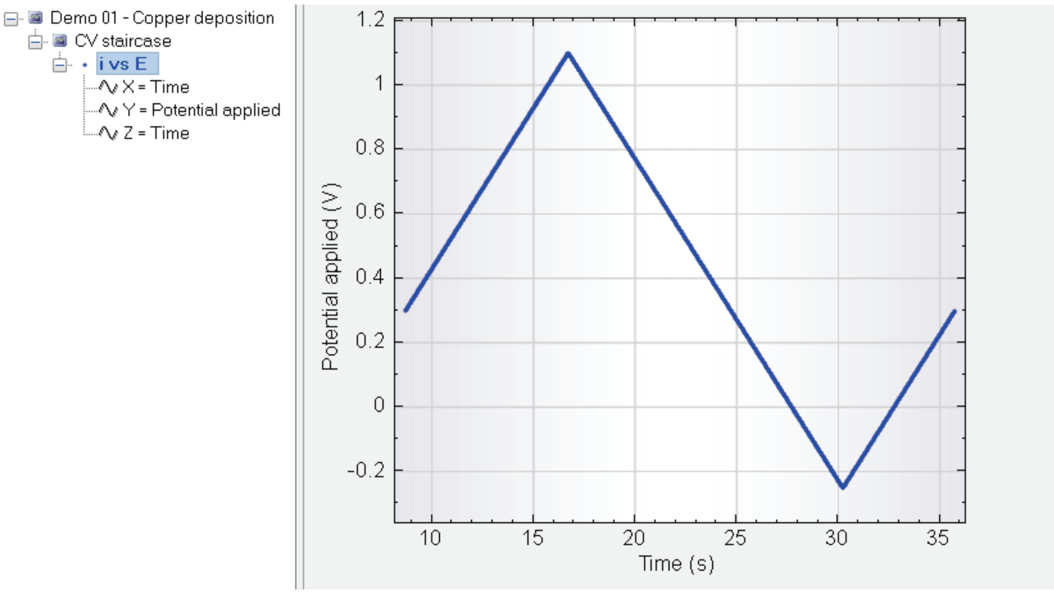


Figure 4.27 – The saw-tooth profile of the cyclic voltammogram of Example #1

Note

Changing the signals used to display the data points from example #1 also changes the labels displayed on the axes of the plot.

Warning

The **time** signal is the total elapsed time since the beginning of the measurement. It includes the preconditioning time used during the experiment. If required, it is possible to use the calculate signal tool to correct the time scale (see section 4.10).

4.2.3 – Changing the plot options

Although NOVA uses default settings for all the plots, it is possible to customize the appearance of each individual plot. This can be done by right-clicking a plot in the data explorer frame and selecting the **Plot Options** item from the context menu (see Figure 4.28).

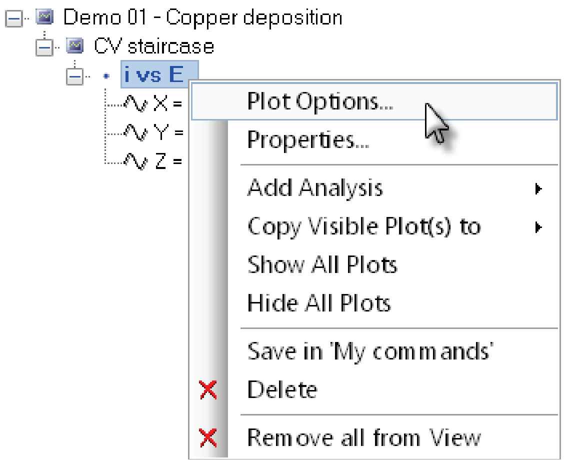


Figure 4.28 – Changing the plot options

The Plot Options window will be displayed. Through this window, it is possible to customize every aspect of the selected plot (see Figure 4.29). It is possible to change the plot color, the plot style, the point markers, axes configuration, etc.

Note

The settings defined in the Plot options window are used for both 2D and 3D plots. It is possible to define specific settings for both data presentation formats by clicking the Advanced button in the Plot options window.

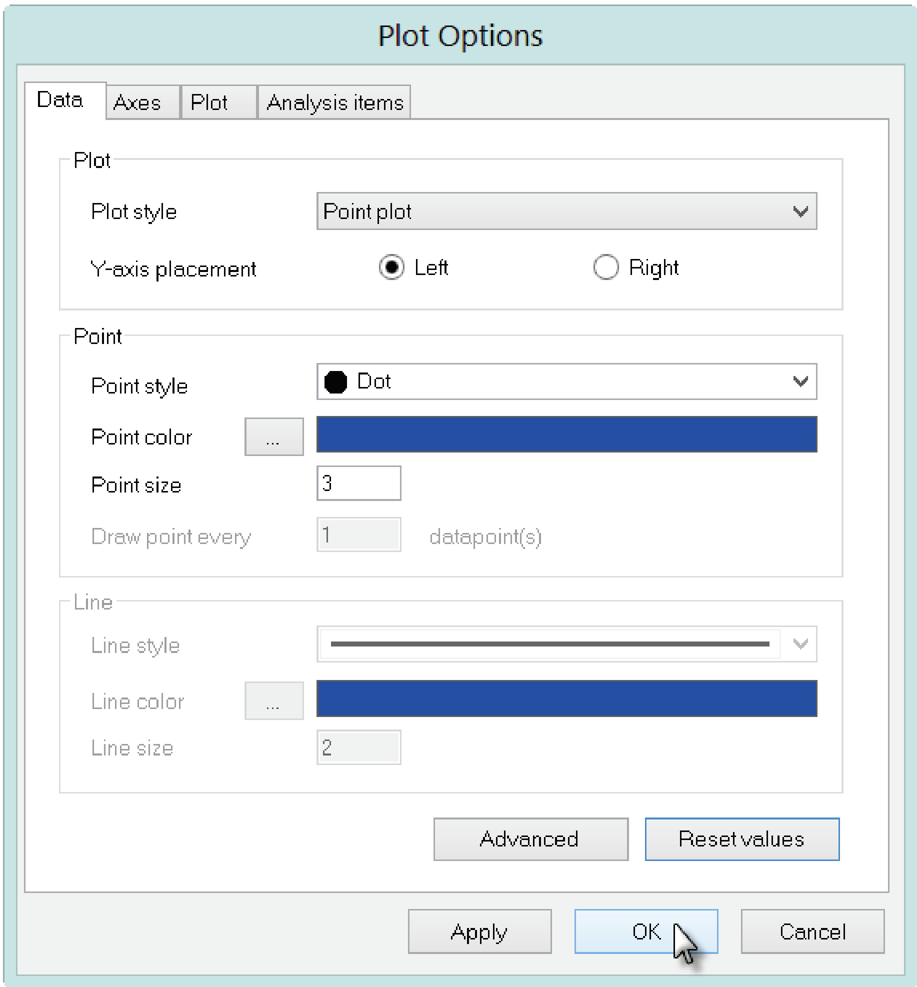


Figure 4.29 – The plot options window

The plot options window has four different **tabs**, which can be used to define different aspects of the plot.

4.2.4 – Data tab

The items located on this tab are related to the plot appearance. It is possible to choose from different plot style, to change the plot color and size and define a point style. It is also possible to change the position of the Y-axis (see Figure 4.29).

4.2.5 – Axes tab

This tab allows changing of the axes-related options like type of scaling and formatting, axes coupling, axes labels. It is also possible to define specific axes labels that will overwrite the defaults signal names in the plots (see Figure 4.30).

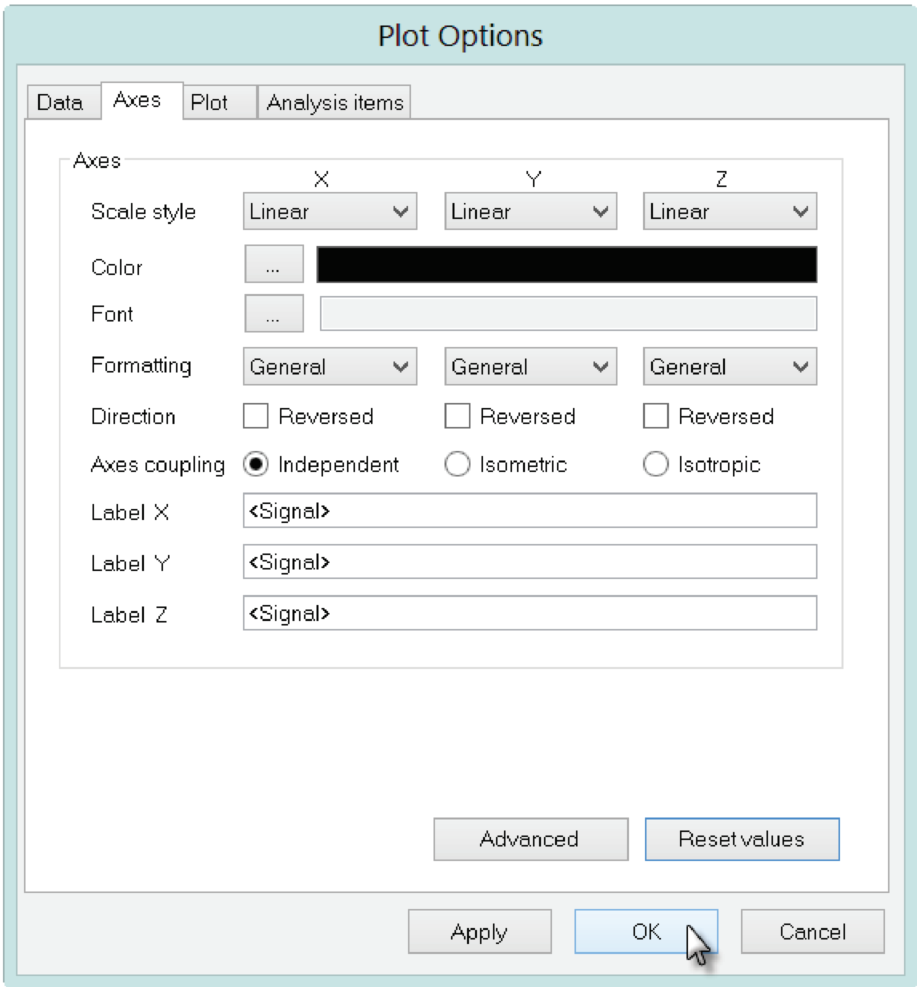


Figure 4.30 – The axes settings tab

4.2.6 – Plot tab

On this tab, it is possible to define a title and a subtitle as well as their format. It is also possible to define the grid settings and switch the legend on or off (see Figure 4.31).

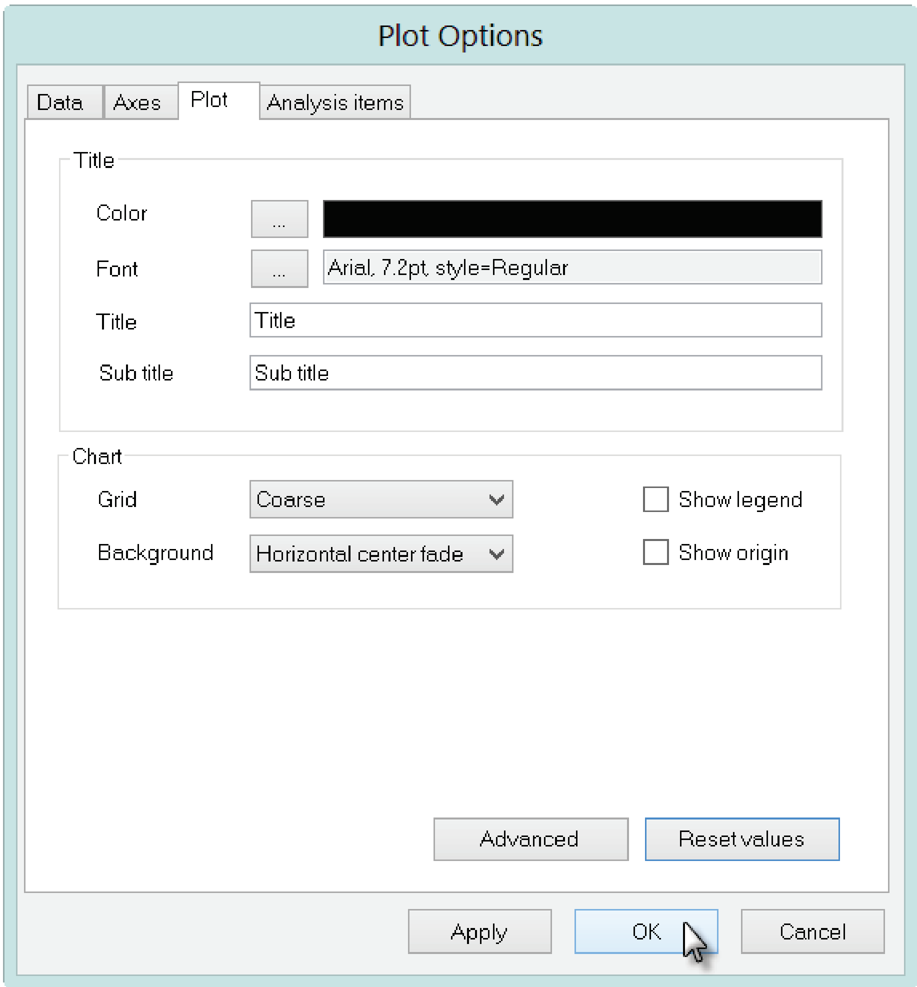


Figure 4.31 – The plot settings tab

4.2.7 – Analysis items tab

This tab defines the plot options used for the graphical data analysis tools. All items added to a plot after data analysis (like linear regression, fit and simulation, etc…, will be displayed using these settings.

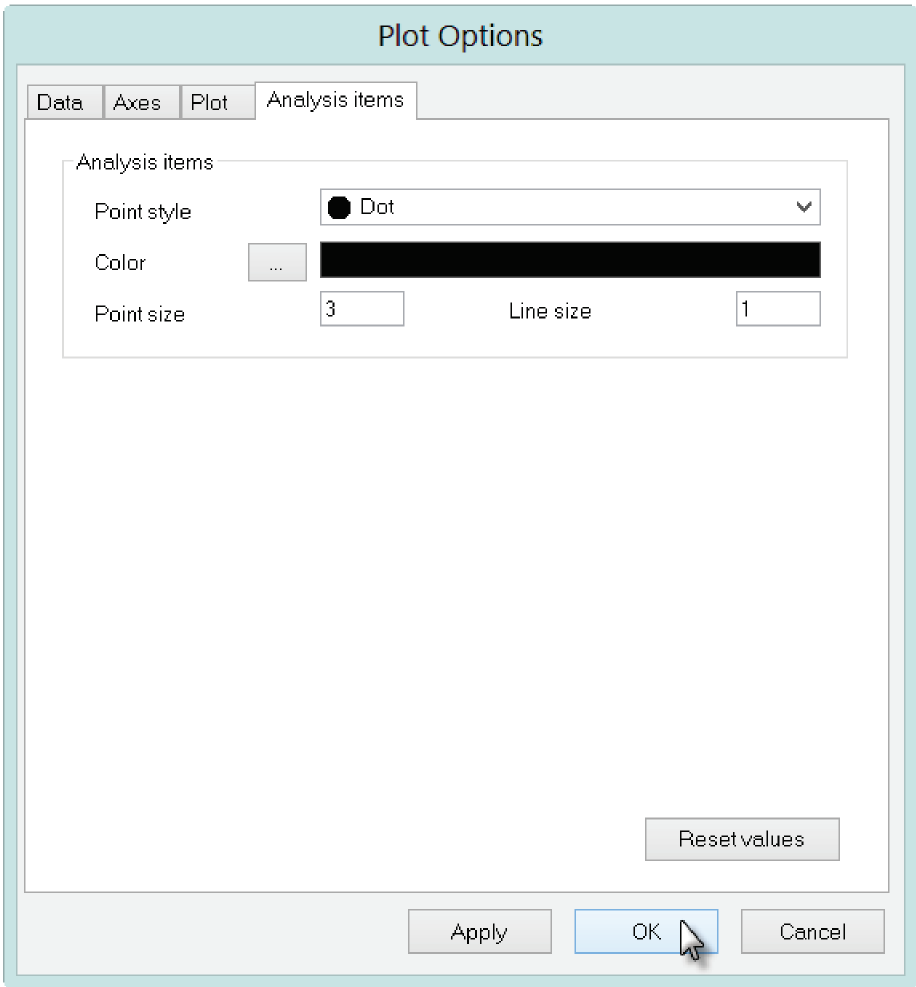


Figure 4.32 – The analysis items tab

The plot options window provides a convenient interface for changing all the available plot settings at the same time. It is still possible to change specific plot options by right-clicking the corresponding items on the plot directly. For example, right-clicking on a plot axis will display a menu from which some of the options available in the Axes settings tab can be defined (see Figure 4.33).

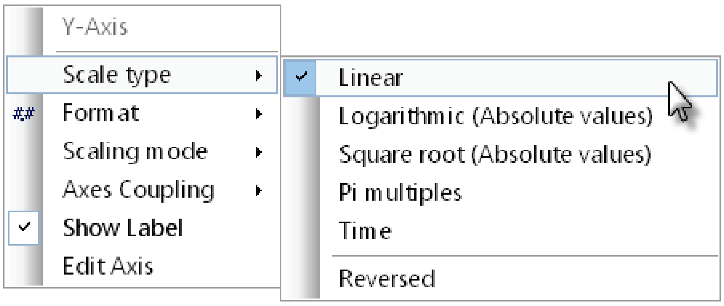


Figure 4.33 – Right-clicking a plot axis displays a context menu

Note

Contrary to the setting defined in the Plot Options window, the settings defined using the context menu are only valid for the related item in the active plot. Changing the Y-axis settings in a 2D plot does not affect the appearance of the corresponding 3D plot.

As an example, select the **i vs E** plot from Example #1 data set and using the Plot Options window, change the plot settings to a red Combi plot, using a size 10 pyramid as a symbol, plotting 1 point out of 40 and using a size 2 line (see Figure 4.34).

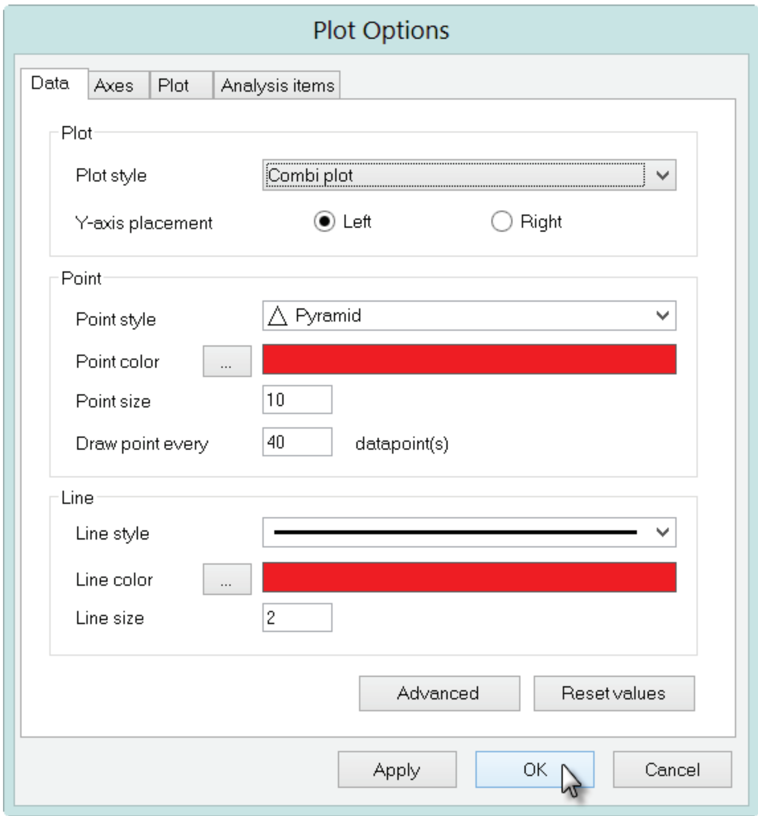


Figure 4.34 – The modified plot options for Example #1

The cyclic voltammetry saw-tooth profile should now be displayed as in Figure 4.35.

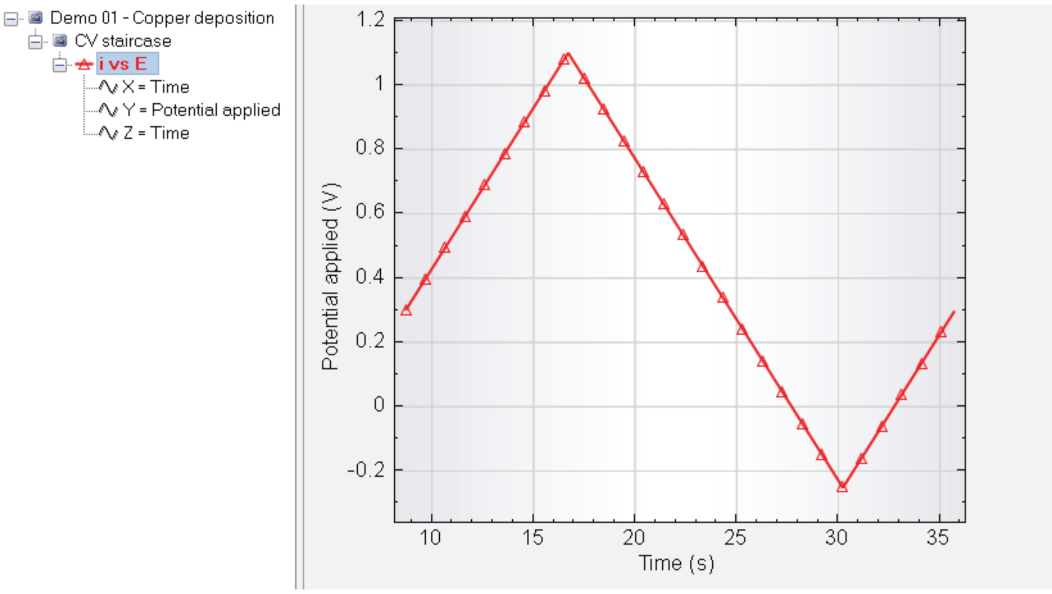


Figure 4.35 – New plot settings for Example #1

Note

The i vs E has changed color and is preceded by a red pyramid symbol (i vs E). This feature makes it easier to identify a specific plot in an overlay (see Section 4.4).

4.2.8 – Creating new plots

It is often convenient to plot experimental data in different ways. NOVA offers this possibility directly from the analysis view. Using the right-click menu, it is possible to add any number of new plots to a data set. Two different types of plots can be created: a command-specific predefined plot or a generic Custom plot. In this section, both options will be illustrated.

NOVA provides a number of predefined plots for every measurement command. The experimental data from Example #1 was measured using the **CV staircase** command, for which a total of nine predefined plots are available. To add any of these plots to the data set, right-click the CV staircase line in the data explorer and select the **Add Plot** option from the context menu.

Nine plots will be provided in an additional sub-menu (see Figure 4.36):

- Custom
- i vs E
- i vs t
- Log(i) vs E
- Log(i) vs Log(t)
- E vs i
- E vs t
- E vs Log (i)
- E vs Log (t)

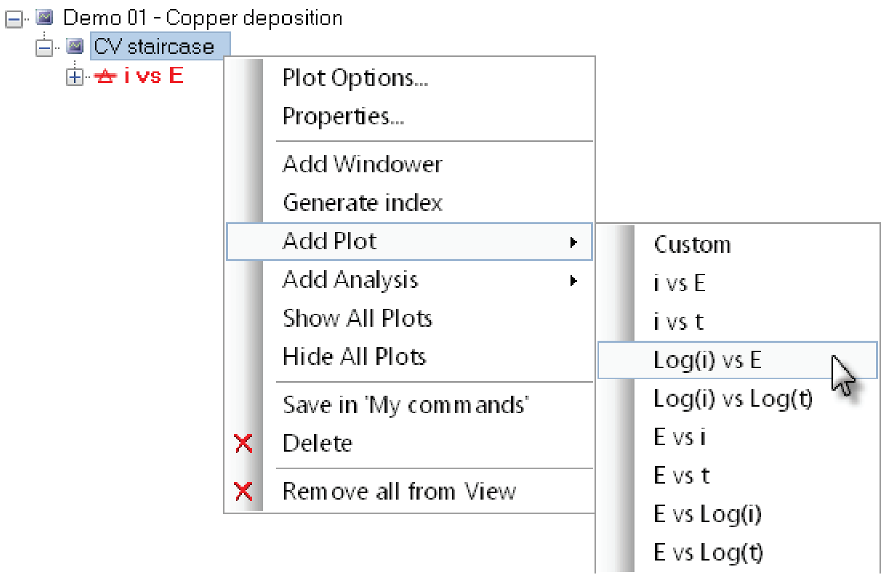


Figure 4.36 – Adding a standard plot to the data set

Note

The number of available plots depends on the signals available in the data set.

It is also possible to add a plot to the measurement by using the quick access toolbar. Clicking the button displays the list of available plots (see Figure 4.37).

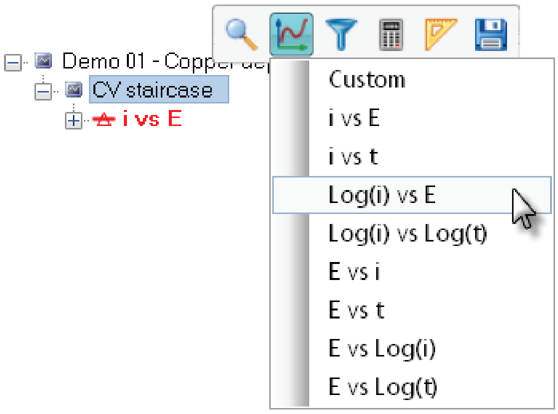


Figure 4.37 – Using the quick access toolbar to add a plot to the data set

Select the Log(i) vs E plot. This will add a predefined plot to the data set, displaying the WE(1).Current on the Y-axis, in logarithmic scaling and the Potential applied on the X-axis. The plot will be displayed using the default plot settings (see Figure 4.38).

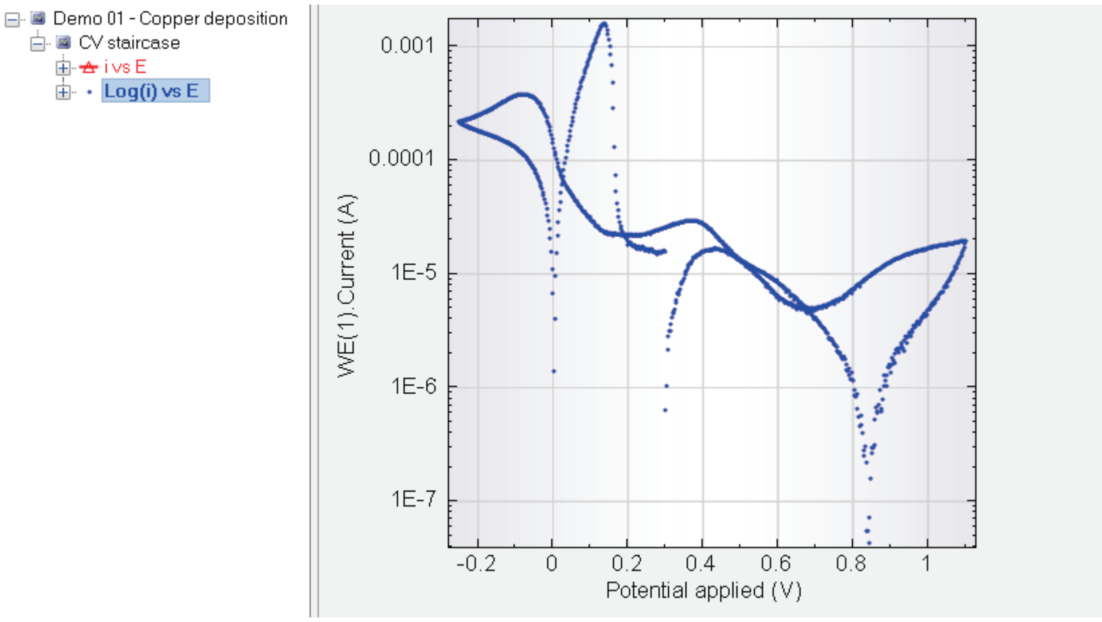


Figure 4.38 – Adding a pre-defined plot to the data set

It is also possible to add a generic new plot to the data set, simply called Custom, which does not have any predefined settings. To do this, right-click the **CV Staircase** line in the data explorer frame and select the **Add Plot** option (see Figure 4.39).

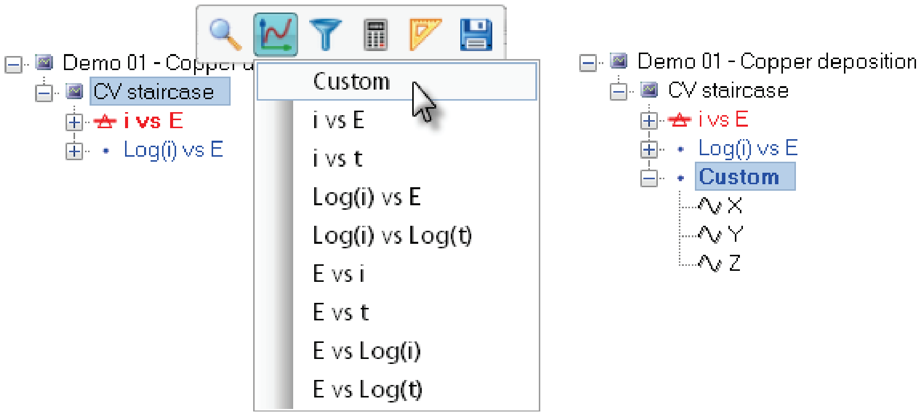


Figure 4.39 – Creating a new plot for the data set

The newly created **Custom** plot does not have any signals assigned for X, Y and Z and therefore does not plot anything until these are defined by the user.

Change the signals in order to plot the measured current relative to the time. (X = Time, Y = WE(1).Current). Change the plot settings to a red Combi plot, using a black pyramid upside down as a symbol, size 10, plotting 1 point out of 40 and using a size 2 line (Figure 4.40).

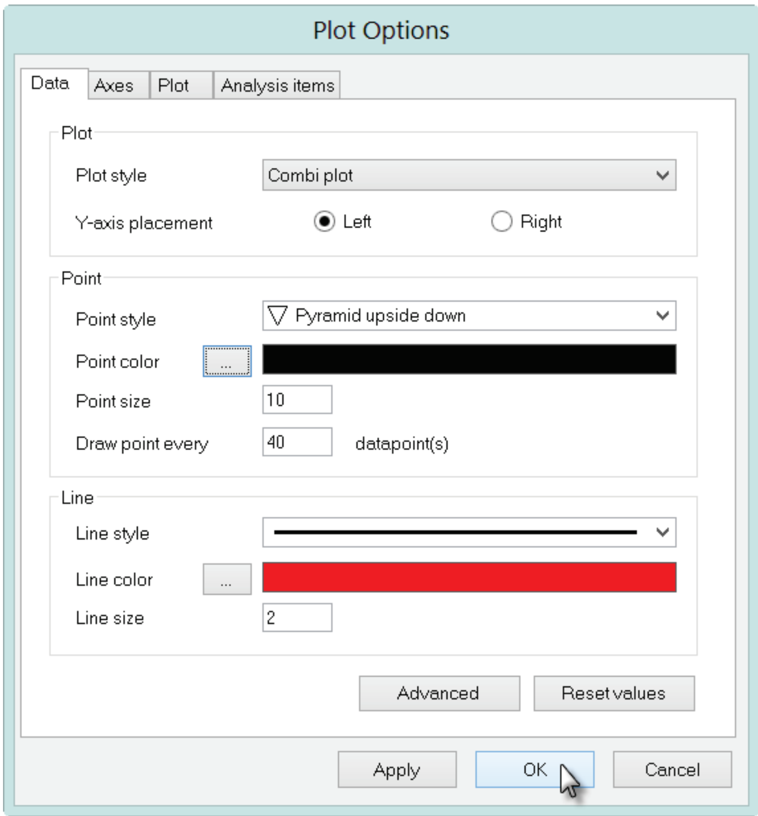


Figure 4.40 – Setting the options for the newly added plot

This new plot should like the one displayed in Figure 4.41.

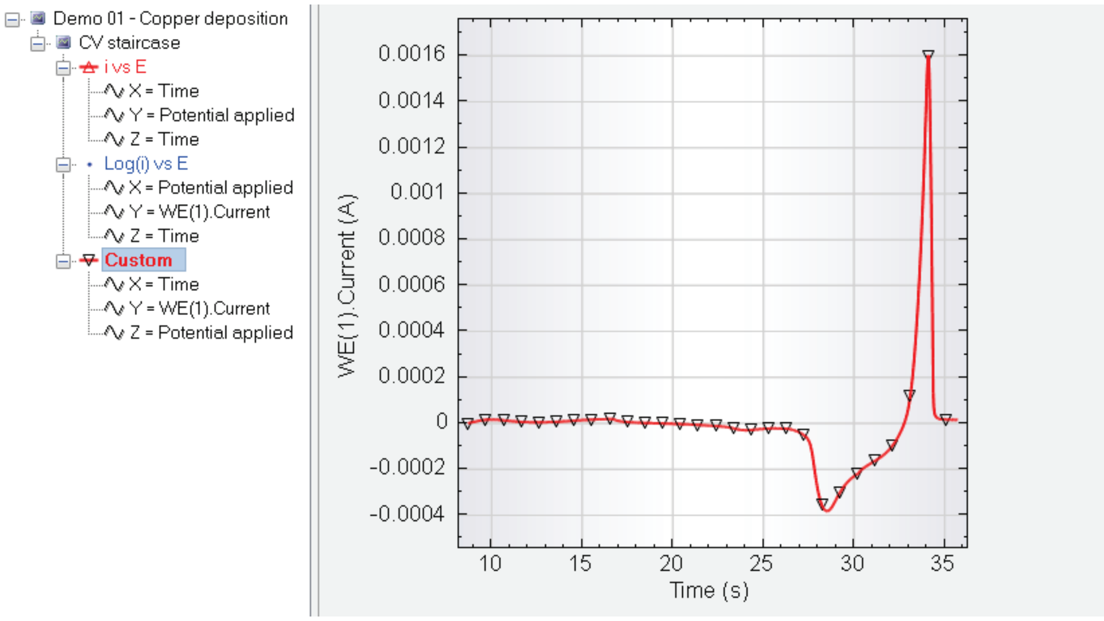


Figure 4.41 – Plotting the current vs. time in a new graph

Each plot can be renamed. To change the name of a plot from Custom to something else, right-click the Custom line you want to edit and select the Properties option. A small window will be displayed which allows you to edit the name of the plot (see Figure 4.42).

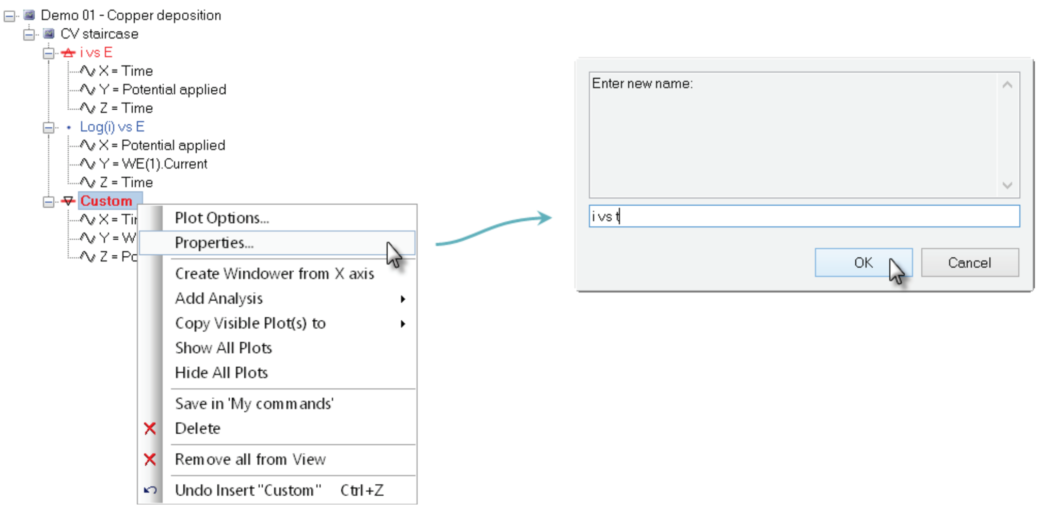


Figure 4.42 – Editing the names of the plots

Rename the first plot *E vs t* and the third plot to *i vs t* (see Figure 4.43).

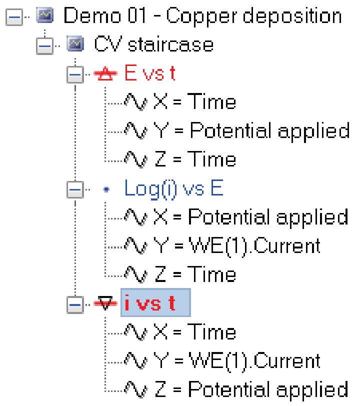


Figure 4.43 – The edited plot names for Example #1

4.2.9 – Saving the changes

The changes that have been made to the plots of Example #1 can be saved in the database. To save these changes, the entry of the database corresponding to the edited data set must be updated.

To save the changes, click the Save button on the data analysis toolbar (see Figure 4.44). This will save the changes of all the open data sets, without changing the time stamp and the remarks.

Figure 4.44 – Updating the database

Note

If the ongoing session of Nova is closed or if the data set is removed from the data explorer frame before the changes have been saved, a warning message will be displayed prompting the user to choose whether or not to update the database. Clicking the Yes button will update the database in the same way as the **Save** button. Choosing No will discard all the changes. A Yes to All/No to All button is also available, for all the open data sets (see Figure 4.45).

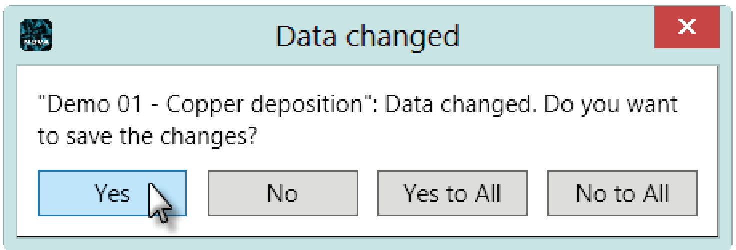


Figure 4.45 – Dataset changed warning message

Note

It is also possible to save the changes to a data set by right-clicking the data set in the data explorer frame and selecting the Save the data in database option (see Figure 4.46).

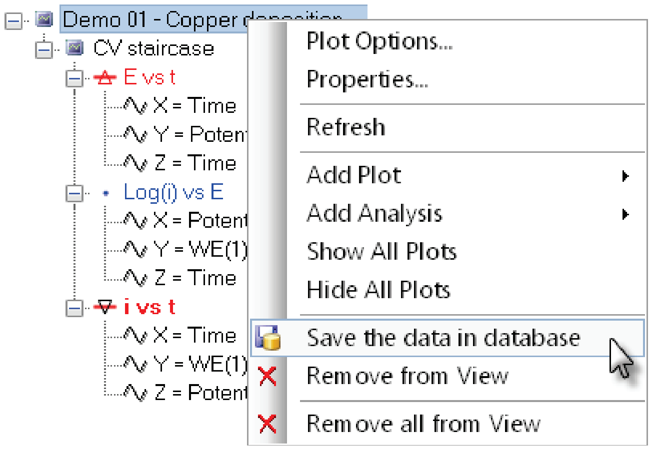


Figure 4.46 – Using the *Save the data in the database* option to update the whole data set

4.3 – Plotting the data in 3D

NOVA introduces a powerful 3D graphic engine that allows the use of three signals to plot the data. The plotting options and settings are common to those of the 2D plots, which means that any change of these settings will affect both types of plots.

Load the data set from Example #1 back into the data explorer (if necessary). Create a new plot, called Standard CV, using a green Line plot, using a size 2 line (refer to the previous sections if required).

Set the Potential applied on the X-axis, WE(1).Current on the Y-axis and Time on the Z-axis (see Figure 4.47).

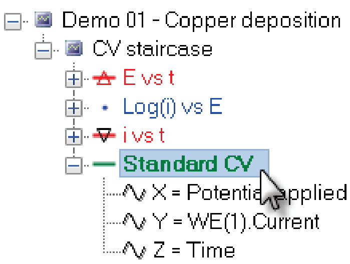


Figure 4.47 – Creating the Standard CV plot

To view this new plot in 3D, click the 3D view button in the Data Analysis Toolbar (see Figure 4.48).

Figure 4.48 – Switching from 2D to 3D

When the 3D button is clicked, NOVA will display the data from example #1 in a perspective, 3D plot (see Figure 4.49).

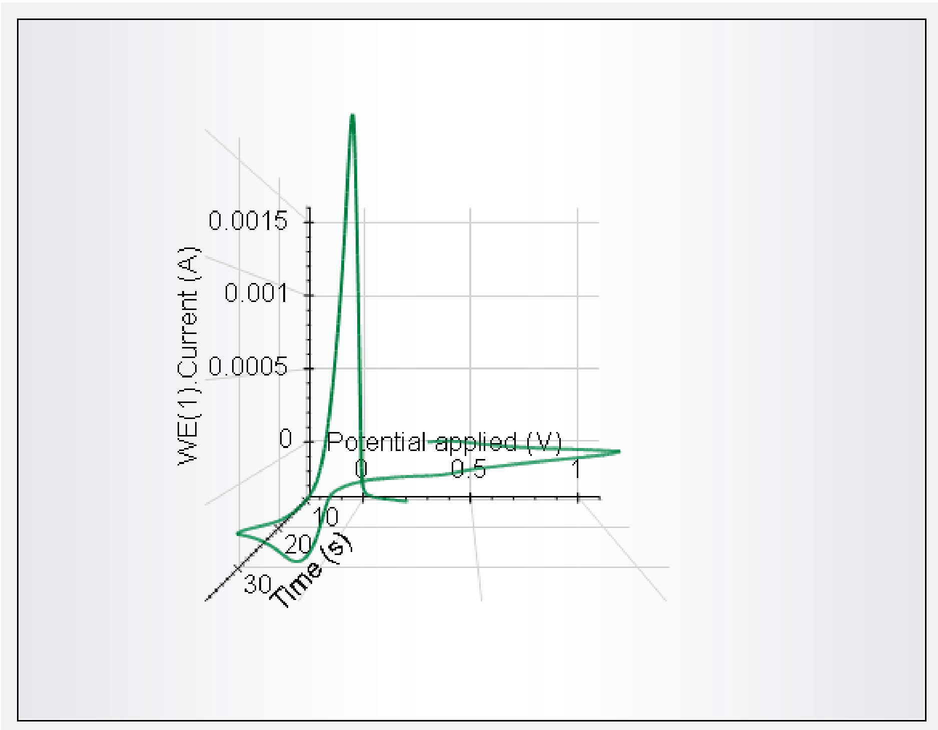


Figure 4.49 – The data from Example #1 in 3D

Press and hold the right arrow key on your keyboard to rotate the 3D plot around the Y-axis, slowly bringing the time axis in plane with your computer screen (see Figure 4.50).

Note

You can use the CTRL key in combination with the arrow key to rotate the plot faster. Press the Page Up and Page Down keys to spin the plot in the X/Y plane.

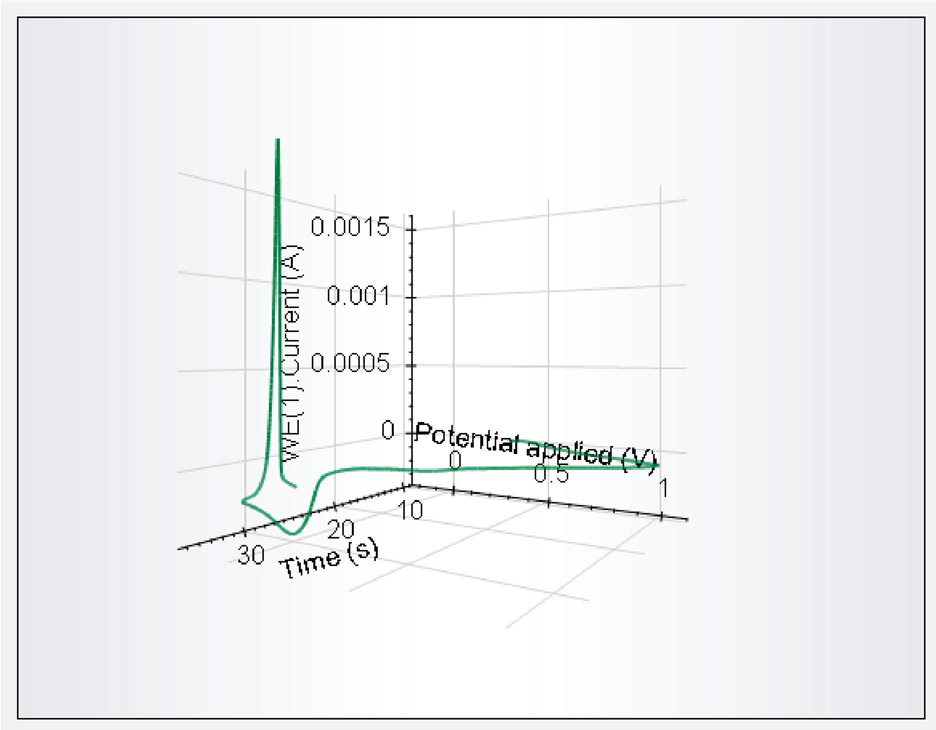


Figure 4.50 – Rotating the 3D view along the Y-axis

Alternatively, click anywhere on the 3D plot, and move the mouse in any direction, *while holding the mouse button*, to spin the plot in the direction of your choice (see Figure 4.51).

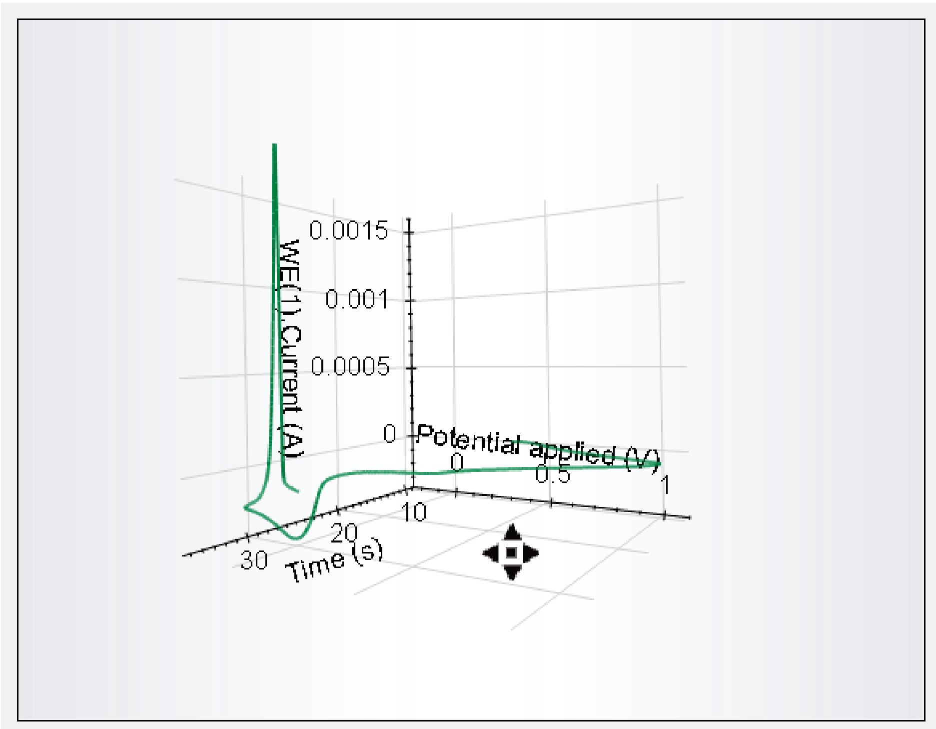


Figure 4.51 – Moving the 3D plot with the help of the mouse pointer

Note

Pressing the F4 function or the Home key will restore the plot to its original size and orientation.

NOVA offers two types of 3D projections to visualize the data. By default, the perspective projection is selected. An alternative orthogonal projection is available (see Figure 4.52).

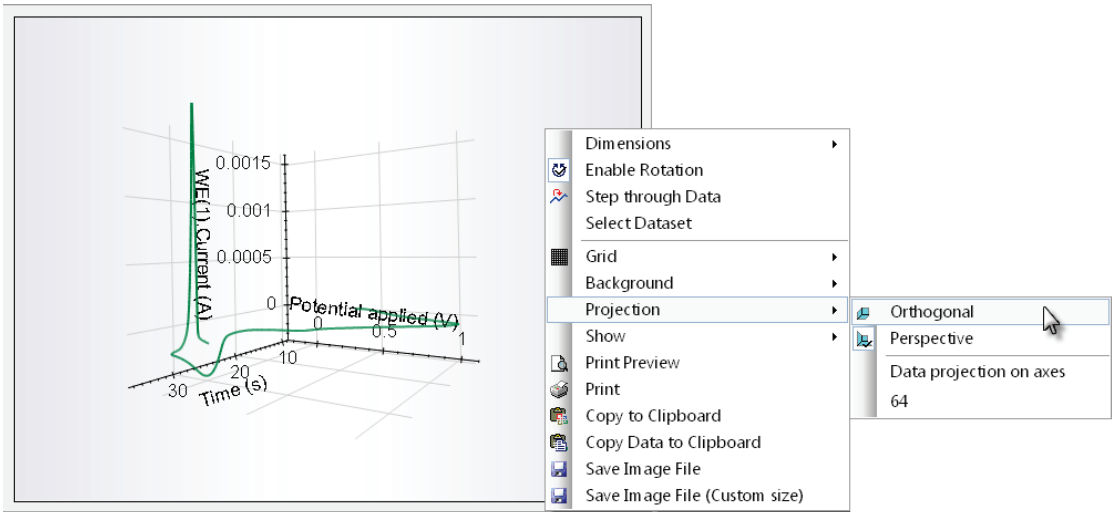


Figure 4.52 – Plotting the data using the orthogonal projection

4.4 – Advanced plotting and overlays

NOVA offers a large number of options that can be used to further customize the data plots and create overlays of different plots. This section explores these options using a new example data set.

Note

The *Demo 02 – Lead deposition EQCM* entry of the demo database contains three cyclic voltammograms recorded using a gold / TiO_2 polished (6 MHz) crystal, in 0.01 M $Pb(ClO_4)_2$ / 0.1 M $HClO_4$ solution. The reference electrode was a solid Ag/AgCl (KCl Sat'd) and the counter electrode was a platinum rod.

Load *Demo 02 - Lead deposition EQCM* in the data explorer frame. The signals available for this data set are:

- Potential applied
- **WE(1).Current**
- WE(1).Potential
- **EQCM(1).ΔFrequency**
- **EQCM(1).Temperature**
- **EQCM(1).Driving force**
- Index
- Scan
- Time

EQCM(1).ΔFrequency is the signal containing the values of the EQCM frequency change recorded using the Autolab EQCM, during the measurement. Both WE(1).Current and EQCM(1).ΔFrequency are potential dependent. In order to plot both relative to the potential, it is necessary to create two different plots, using the same approach as the one illustrated in the previous section.

A plot, i vs E, is already attached to the CV staircase (see Figure 4.53).

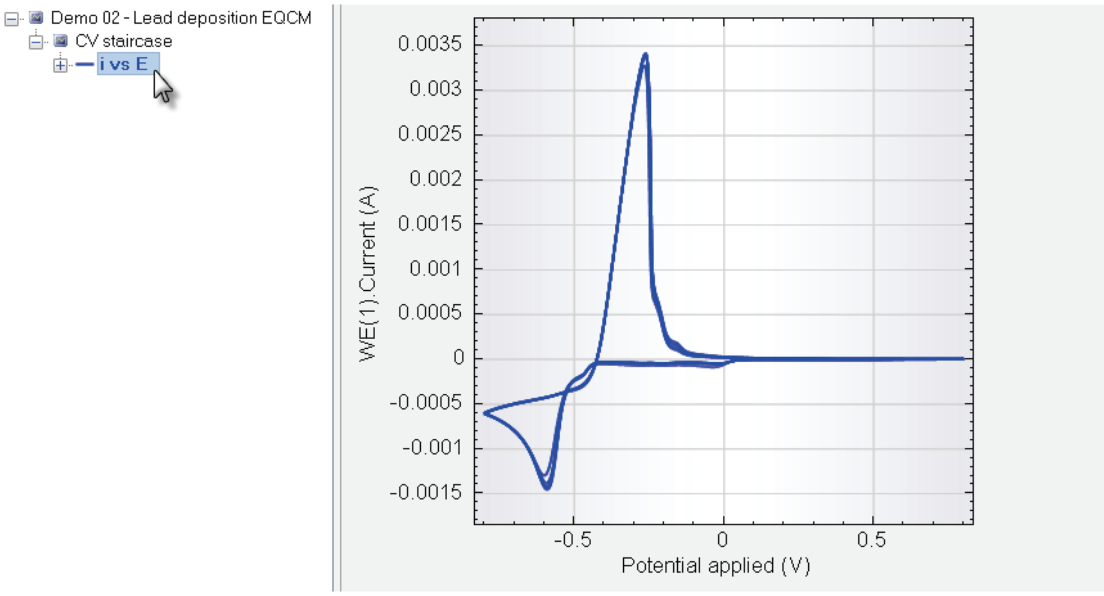


Figure 4.53 – The i vs E plot for Example #2

To view the EQCM(1).ΔFrequency signal versus the applied potential, right click the CV staircase line in the data explorer and choose the ΔFrequency vs E from the Add Plot context menu (see Figure 4.54).

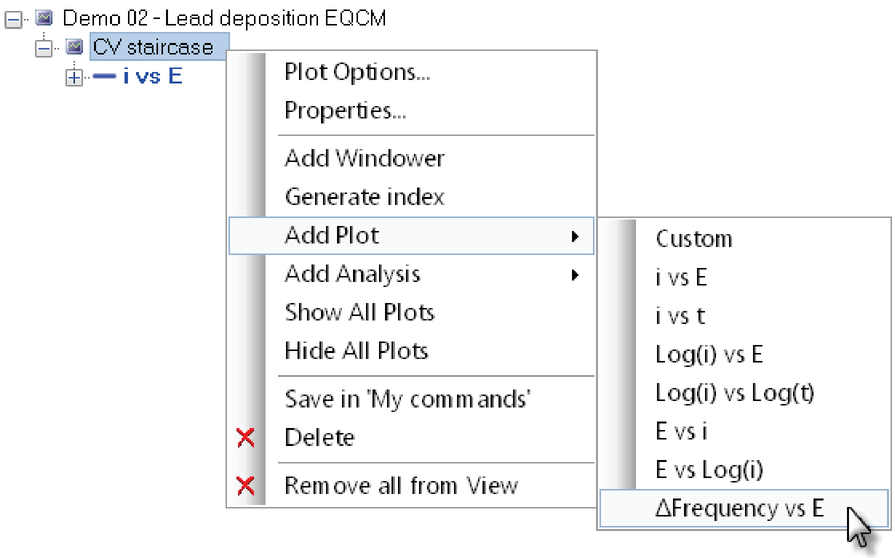


Figure 4.54 – Adding the ΔFrequency vs E plot to the data set

It is also possible to add the ΔFrequency vs E plot to the CV staircase using the quick access toolbar (see Figure 4.55).

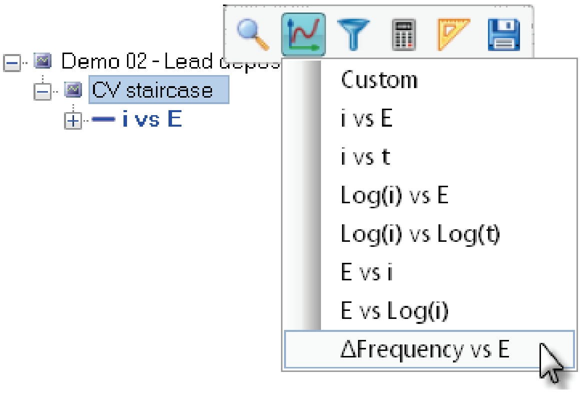


Figure 4.55 – Adding the ΔFrequency vs E plot using the quick access toolbar

You should have two completely different plots at your disposal, shown in Figure 4.56.

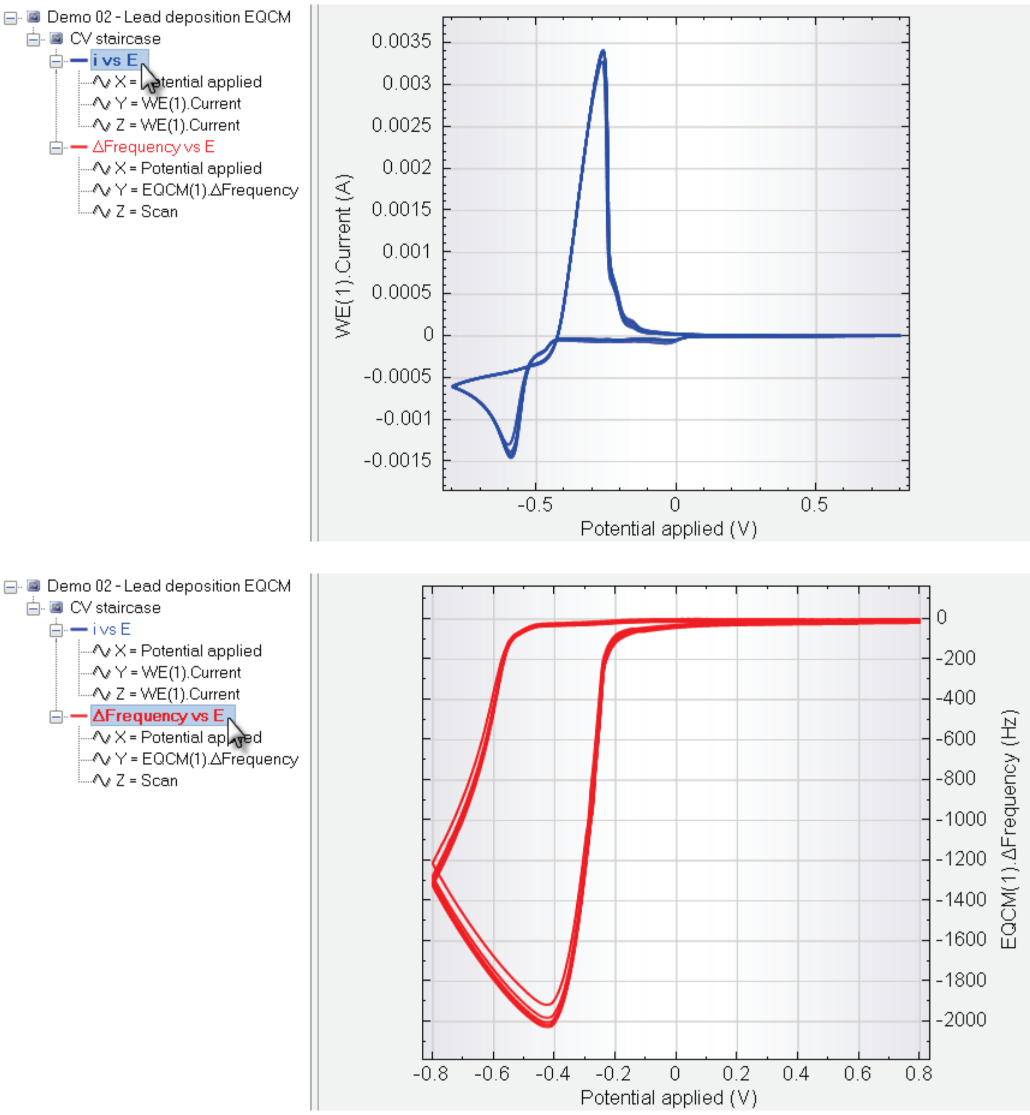


Figure 4.56 – The two plots for the data set from example #2: the measured current relative to the applied potential (top) and the EQCM frequency change relative to the applied potential (bottom)

The data explorer frame should now look like the one shown in Figure 4.57.

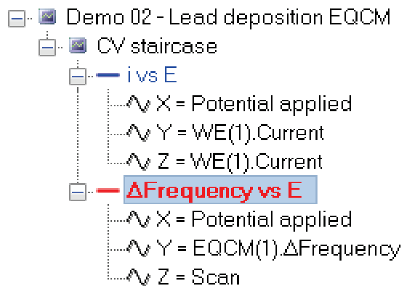


Figure 4.57 – Available plots in the data explorer frame

Presently, only the **ΔFrequency vs E** is being plotted in the 2D plot, indicated by the bold lettering.

To overlay this plot with the i vs E plot, hold the **CTRL** key pressed on the keyboard and click the i vs E plot. The new 2D plot should now look like the one displayed in Figure 4.58.

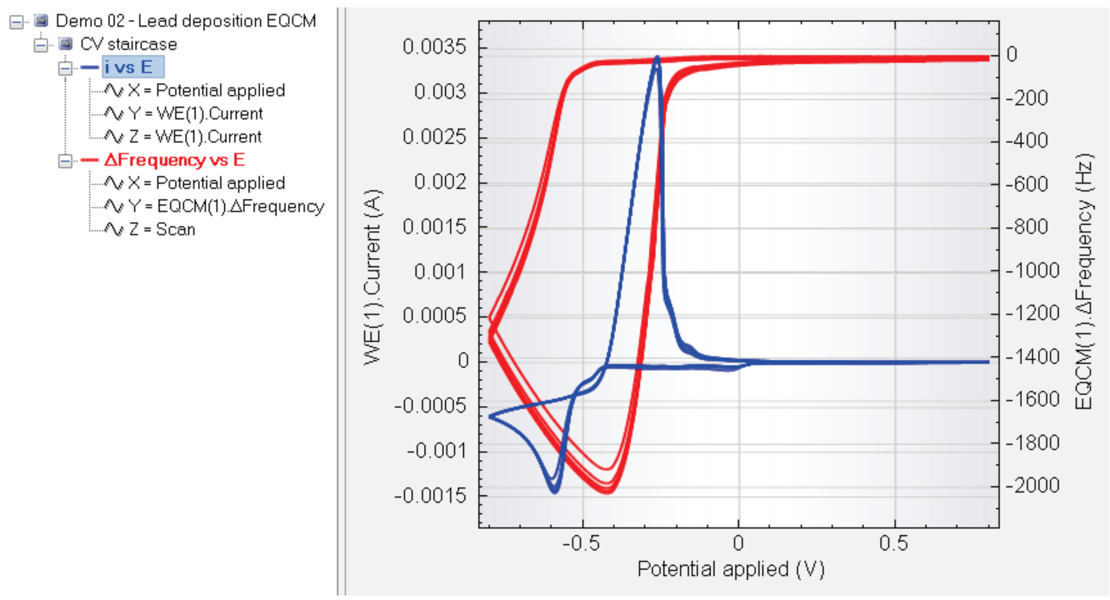


Figure 4.58 – Overlaying both cyclic voltammograms

Note

In the data explorer frame, both the **ΔFrequency vs E** and the **i vs E** plot lines are displayed in **bold** lettering, indicating that both plots are shown in the data view frame. However, only the latter is highlighted, indicating that the **i vs E** plot is currently active. Clicking the **ΔFrequency vs E** line will highlight it, thus setting it to active.

To remove a plot from the overlay, hold the CTRL key pressed and click the line in bold lettering in the data explorer corresponding to the plot you wish to remove.

4.5 – Overlays with the Measurement view

It is possible to create an overlay of the data shown in the Analysis view and the data points recorded in the Measurement view, at any time. This feature allows a quick and convenient comparison between measurements. Up to four different locations can be used to create the overlay in the Measurement view.

To add a plot from the Analysis view to any of the four available plots of the Measurement, right-click the plot in the data explorer frame and select the location of the plot in the Measurement view from the *Copy Visible Plot(s) to* context menu (see Figure 4.59).

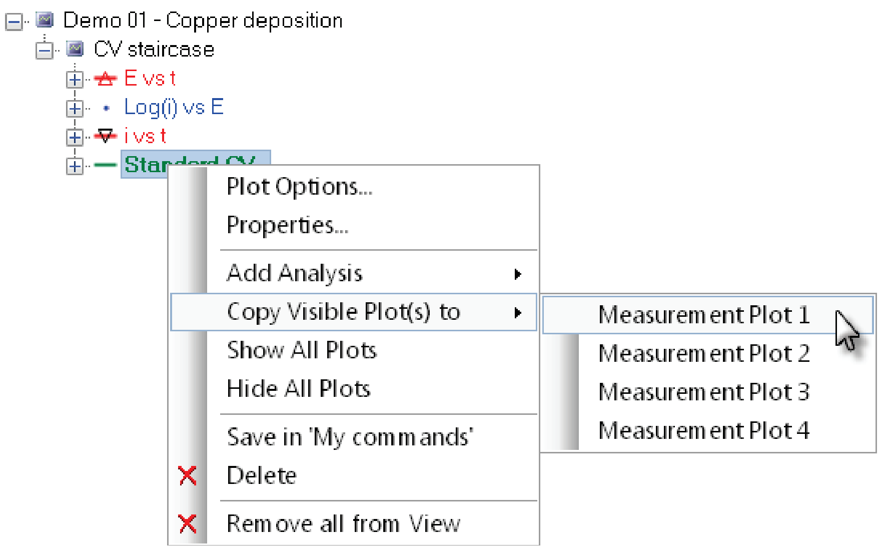


Figure 4.59 – Creating an overlay on the Measurement view using data plotted in the Analysis view

The data shown in the analysis view will be copied as it is displayed to the location specified in the measurement view. If more than one plot is shown in analysis view, all the visible plots will be sent to the measurement view.

Note

The overlay is only shown if the plot is visible in the measurement view. Click the , or button in the toolbar to display more than one plot in the measurement view.

Warning

Overlays created this way must have the same signal on the X axis.

4.6 – Data sets with multiple entries

Some data sets can contain several measurements (if the measurements where performed using a repeat loop for example). While these data sets are managed in the same way as regular data sets, there are some particular features that will be explained in this section.

> **Note**
>
> The *Demo 05 – Fe(II) – Fe(III) on pcPt* entry of the demo database contains four linear sweep voltammograms, in which four different scan rates were used. This measurement is a typical reversible $K_4Fe(CN)_6$ – $K_3Fe(CN)_6$ reaction recorded using a polycrystalline platinum electrode, in 0.2 M NaOH solution [Fe^{2+}] = [Fe^{3+}] = 0.05 M). The reference electrode was a Ag/AgCl (KCl Sat'd) and the counter electrode was a platinum rod.

Load the Demo 05 data set into the data explorer frame. You will notice that the data set contains four different entries (see Figure 4.60)

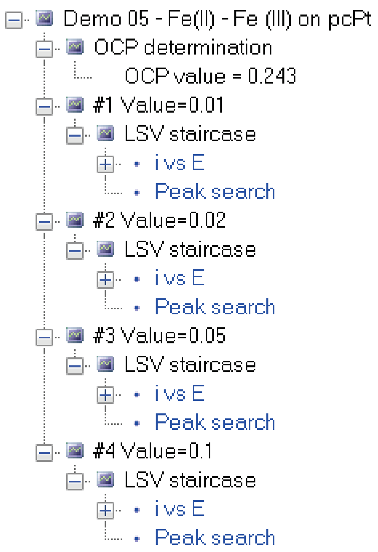


Figure 4.60 – The Demo 05 data set

This data set was obtained using the **Repeat for each value** command, with four values used in procedure for the scan rate of the LSV staircase. The first LSV was obtained with a scan rate of 0.01 V/s, the next LSV was recorded with a scan rate of 0.020 V/s, and so on.

Note

The procedure used to measure the experimental data contains an automatic peak search command. Therefore, each entry of the data set has a peak search analysis item.

Hold the **CTRL** key on the keyboard and click each individual entry of the data set to create an overlay of the four cyclic voltammograms. Since the Fe(II) – Fe(III) electron transfer reaction is reversible, the peak position is independent of the scan rate used in the experiment. The peak current, however, increases with increasing scan rate. Figure 4.61 shows the overlay plot.

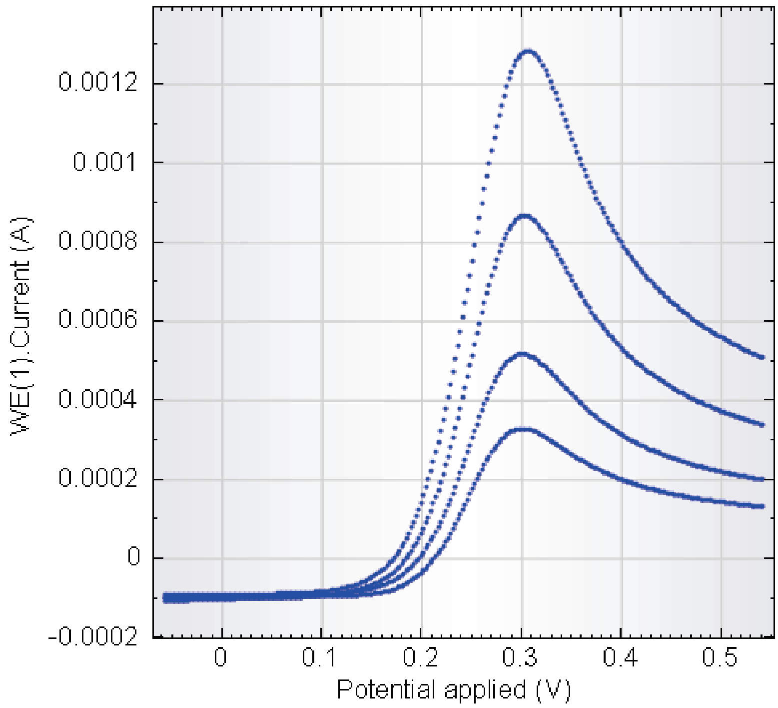


Figure 4.61 – The overlay plot of the four linear sweep voltammetry curves

4.6.1 – Changing the plot settings

To change the plot settings of the overlay displayed in Figure 4.61, the plot settings of each individual plot can be modified, as it was shown in the previous sections. It is also possible to change the plot settings of the whole data set. To modify the whole data set, right-click the Demo 05 header line in the data explorer window and select the Plot Options for the whole data set (see Figure 4.62).

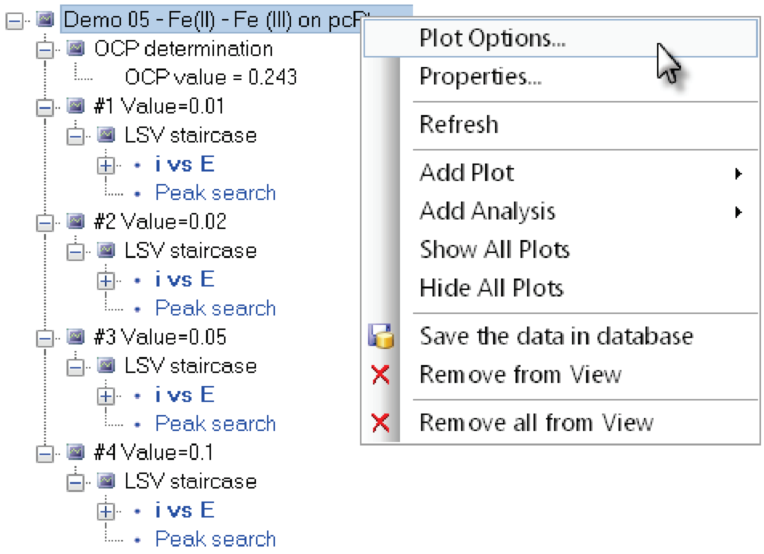


Figure 4.62 – Opening the plot options window for the whole data set

Set the plot style to a size 2, red Line plot. Click the OK button to confirm the change. The four LSV curves will now be displayed using the new plot options (see Figure 4.63).

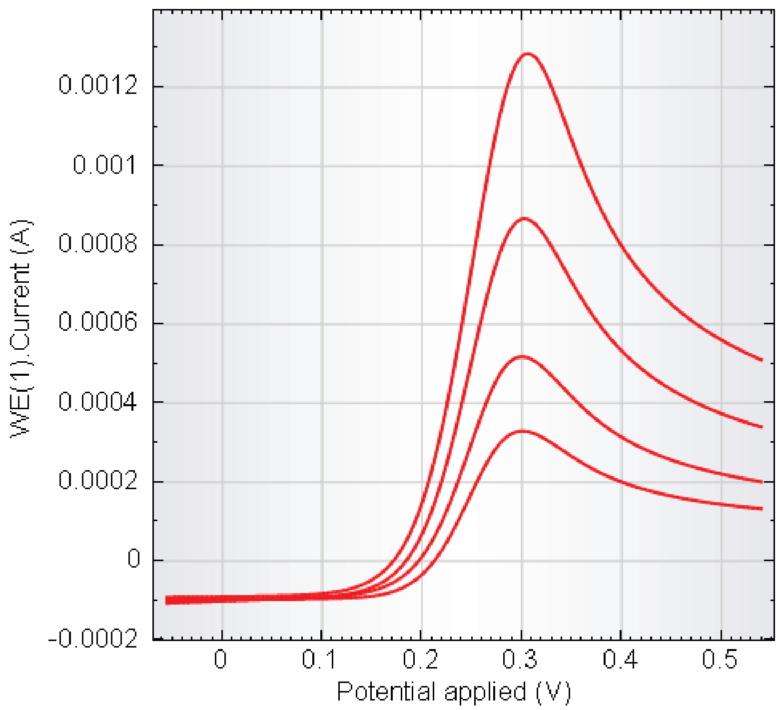


Figure 4.63 – The four LSV plots displayed using the new plot options

Changing the plot settings of an individual plot is still possible. Right-click the last LSV staircase of the data set (0.1 V/s) in the data explorer frame to access the plot options of that particular plot. Change the settings to a size 2, blue Combi plot, using a size 10 star marker symbol and plotting 1 point out of 40. The overlaid plot should now look like the one displayed in Figure 4.64.

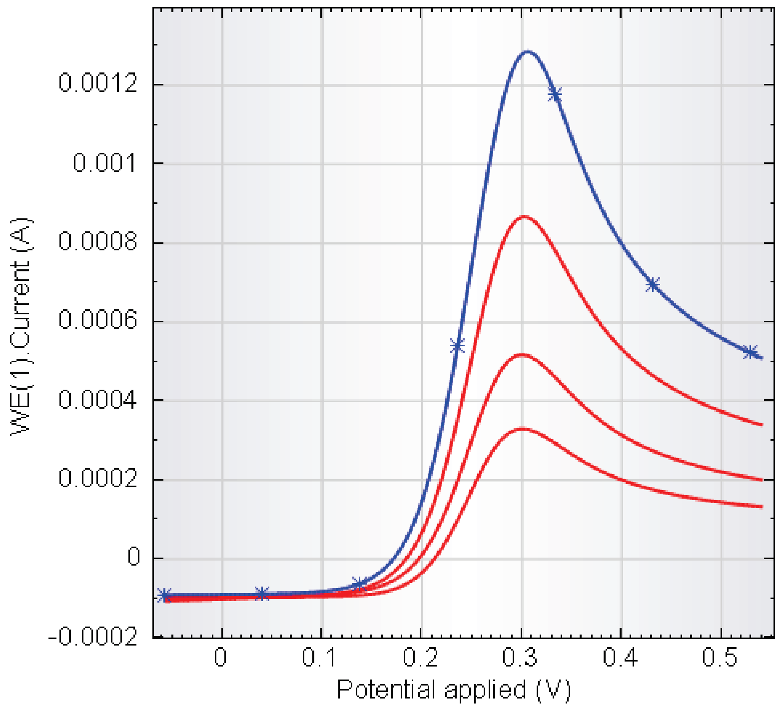


Figure 4.64 – Changing the plot options for the linear sweep voltammetry recorded at 0.1 V/s

4.6.2 – Global options vs Local options

Changing the Plot options for the whole data set defines the plot(s) global options. The local options are defined as the plot options for a specific entry of the data set.

In the example shown in Figure 4.64, the global and local settings are:

- Global options: Red Line plot (size 2)
- Local options (0.1 V/s): Blue Combi plot (size 2), Star marker (size 10, 1 point out of 40)

By design, local options always **overrule** global options. Right-click the Demo 05 line to change the global options of the overlay plot. Set the plot to a black, size 2, Line plot. The resulting plot is displayed in Figure 4.65.

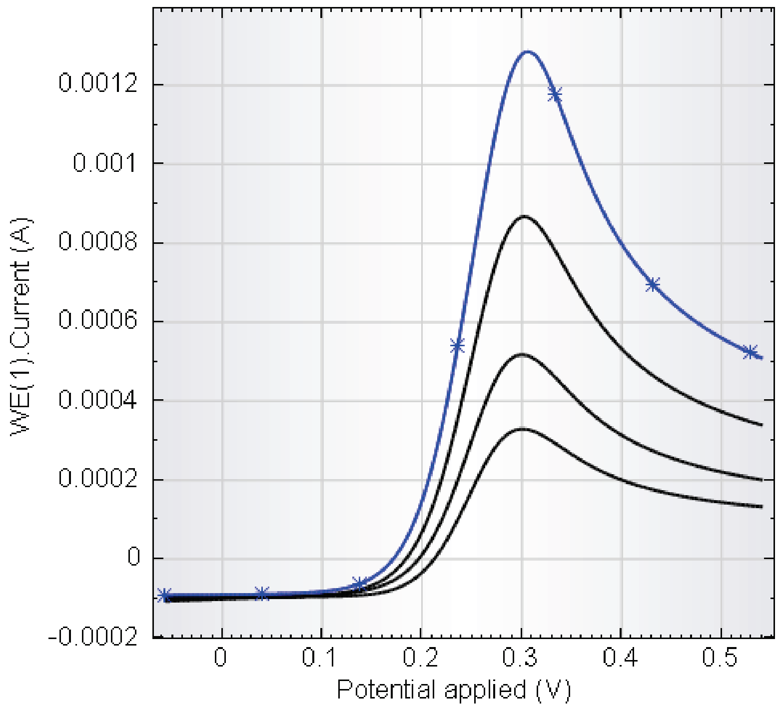


Figure 4.65 – Modifying the global settings of the overlay plot

Changing the plot options for the whole data set only modifies the options for the plots which do not have user-defined local options (in this case, the plot corresponding to a scan rate of 0.1 V/s is not affected).

To remove the user-defined local options, open the Plot options window for the 0.1 V/s plot (use the right-click procedure), and press the Reset values button (see Figure 4.66).

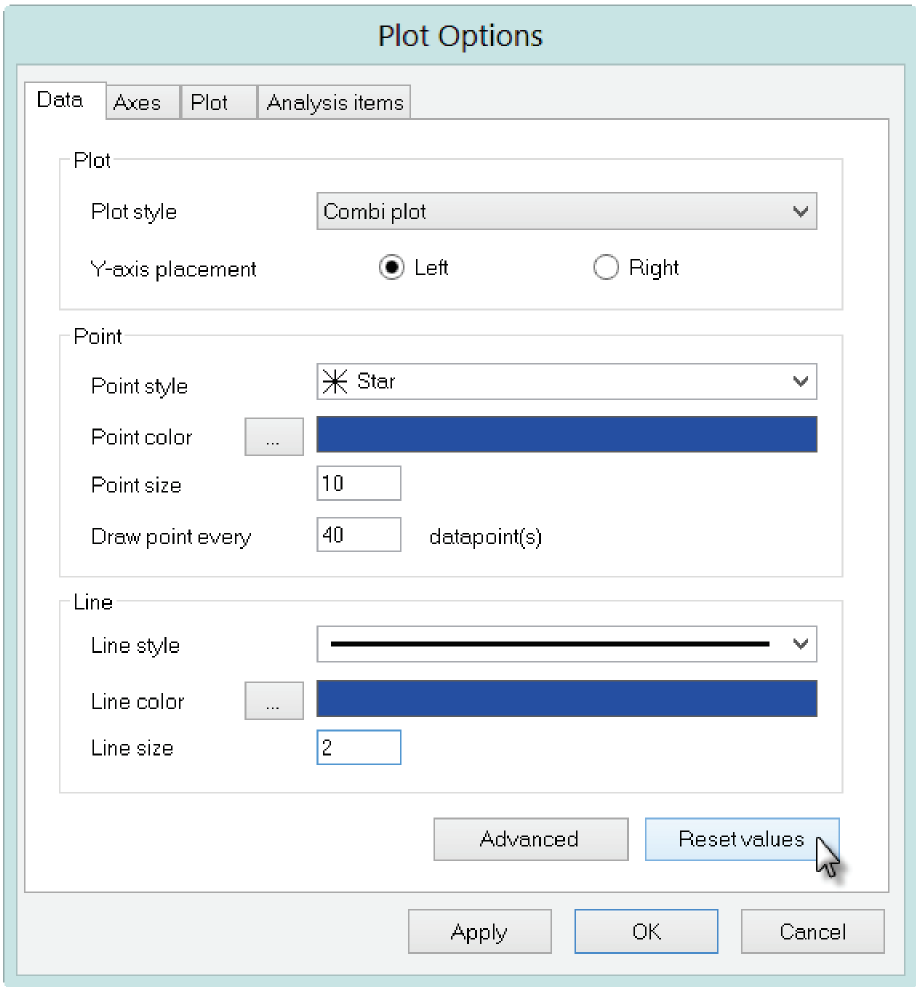


Figure 4.66 – Resetting the plot options for the 0.1 V/s plot (1/2)

Pressing the 

button will remove the local options of the plot and the global options will be used instead (see Figure 4.67).

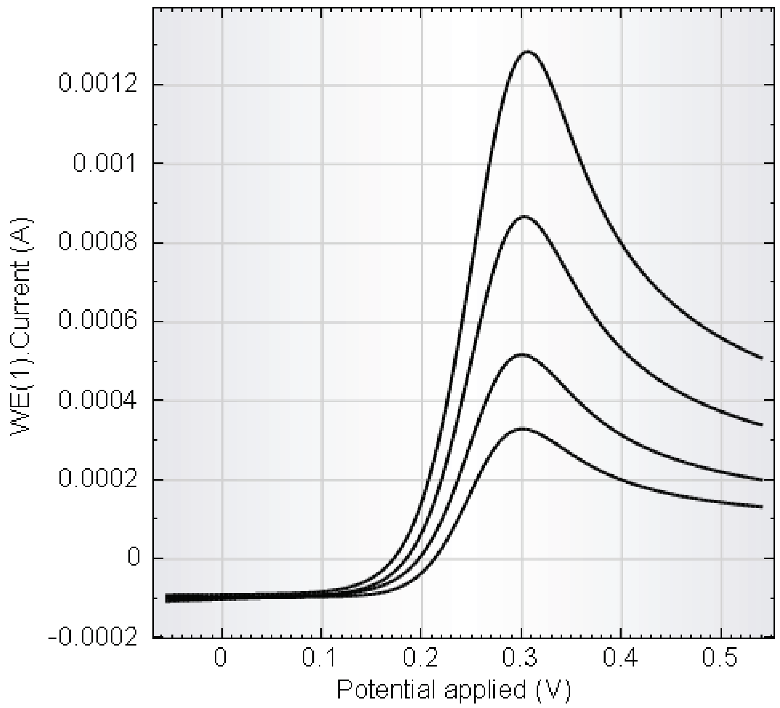


Figure 4.67 – Resetting the plot options for the 0.1 V/s plot (2/2)

If the global options are changed again, all the plots in the overlay will be displayed using these new options, including the 0.1 V/s plot.

4.6.3 – Changing the plot settings in an overlay plot

The curves shown in Figure 4.67 are all displayed as the WE(1).Current vs the Potential applied. This means that all the curves in the overlay are plotted using the same electrochemical signals. If it is required to change the electrochemical signals used to display the curves in the overlay, two options are available.

The first option is to change the electrochemical signals used to display each curve, one after the other. This is quite time consuming, especially for overlays with a large number of plots.

The second option is to change the electrochemical signals of all of the plots of the overlay, by using the *Apply to selected* option (see Figure 4.68). This option can be accessed by right-clicking the signal to change and click the small arrow next of the new electrochemical signal to use.

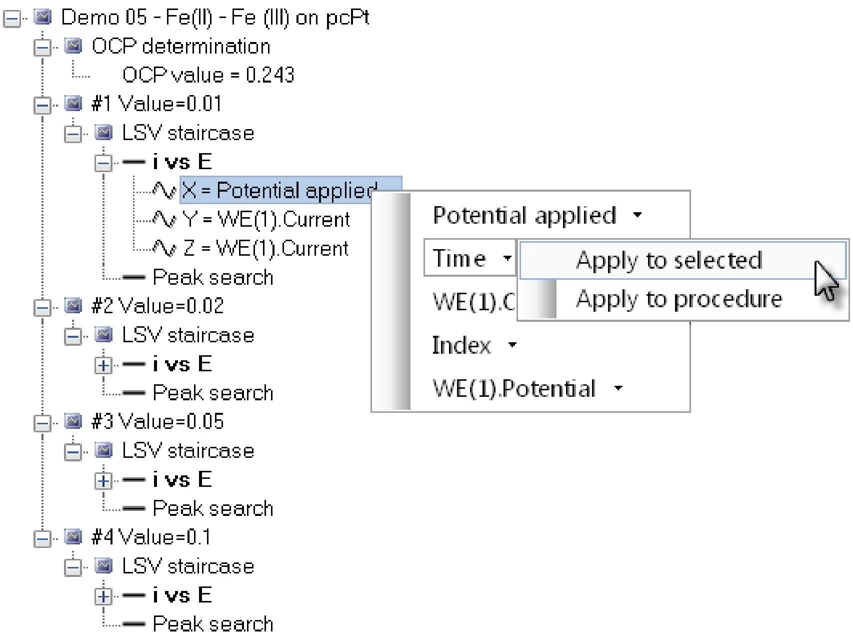


Figure 4.68 – Using the Apply to selected option to change the plot setting of all the plots in the overlay

If this option is used to change the electrochemical signal used on the X-axis for the overlay to the time signal, the resulting plot will look like the one displayed in Figure 4.69.

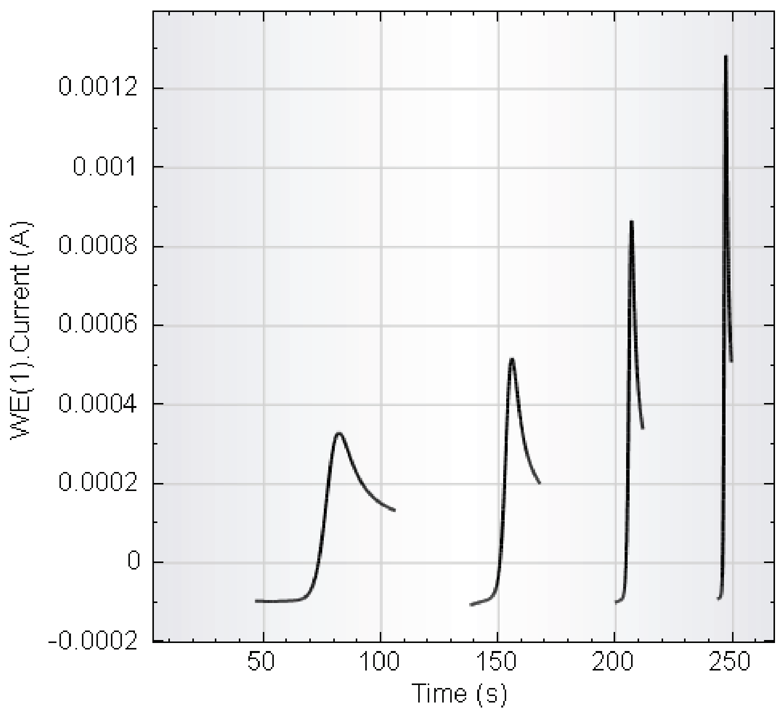


Figure 4.69 – Changing the plot settings for all the plots in the overlay using the apply to selected option

The plot shows the four linear sweep voltammetry curves, obtained with increasing scan rate. The time displayed on the X-axis is the total experiment time. The current increases with each new scan.

Note

The electrochemical signal used on the X-axis has been changed to Time for all the plots in the overlay.

4.7 – Plot objects

Using the 2D/3D plotting feature of NOVA is not limited to just plotting the data. It is also designed to enable NOVA users to prepare high resolution graphs that can be pasted directly into a manuscript or a presentation.

This section of the user manual will explore most of the plotting options related to the layout of the plot rather than to the data itself. The data sets from examples #1 and #2 will be used for illustration purposes.

4.7.1 – The grid

For plotting convenience, a gray background grid is displayed on the plot, making it easier to find a specific point. Using the right-click menu, the grid can be set to fine, coarse or can be turned off (see Figure 4.70). Using a fine grid, horizontal and vertical lines will be plotted for each major and minor tick on the axes. A coarse grid will only plot these lines for the major ticks.

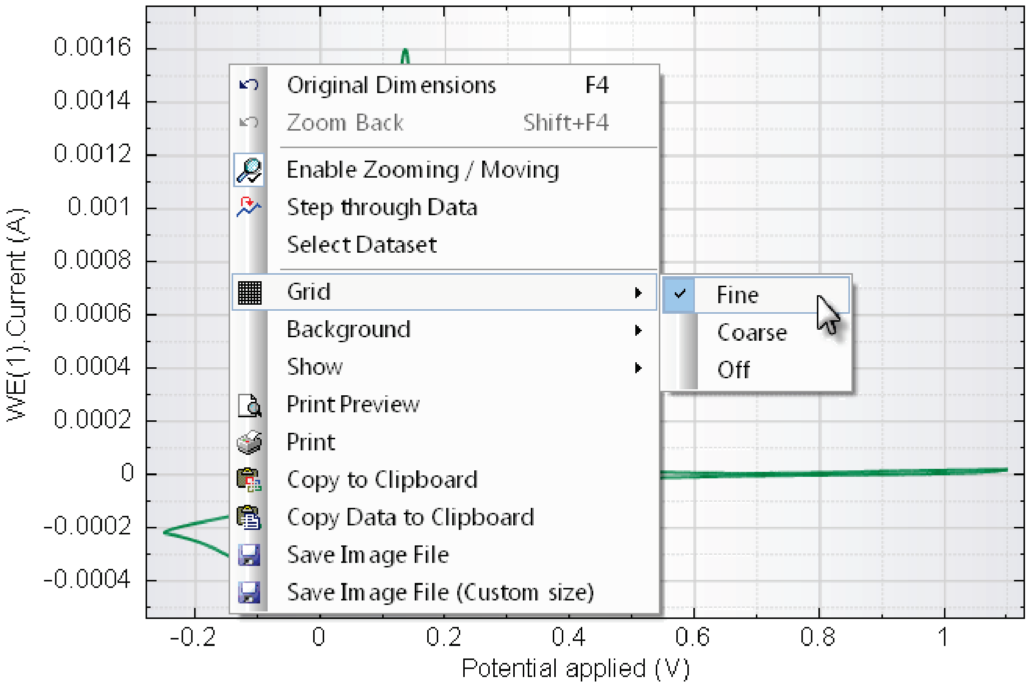


Figure 4.70 – The grid settings can be adjusted using the right-click menu

Note

To avoid overloading the computer, a fine grid used in a 3D plot will temporarily be set to a coarse grid during free mouse rotation of the plot.

4.7.2 – The background

All the plots in NOVA have a default background style (default style: horizontal center fade). The background style can be changed by right-clicking anywhere on the plot and choosing the required background style from the context menu (see Figure 4.71).

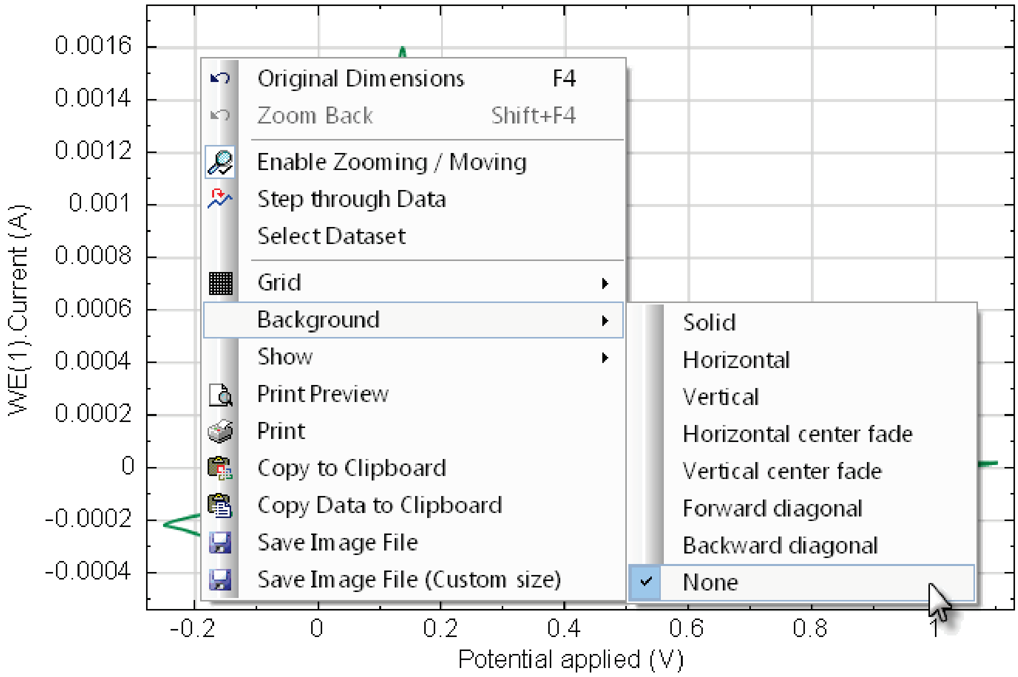


Figure 4.71 – The background style of NOVA can be changed using the dedicated right-click menu item

4.7.3 – Adding objects to the plot

Typical additions to a plot consist of a legend, a title or subtitle, clear axes labels and some markers to indicate the position of relevant points in the plot. NOVA can add these features at any time by using the right-click menu.

Load the data set from example #1 into the data explorer and plot the third plot, called Standard CV plot (restore the Y-axis to its original position). This plot should appear as a green Line plot.

To insert some of the above mentioned additions to the plot, right-click anywhere on the plot. This will display the menu shown in Figure 4.72.

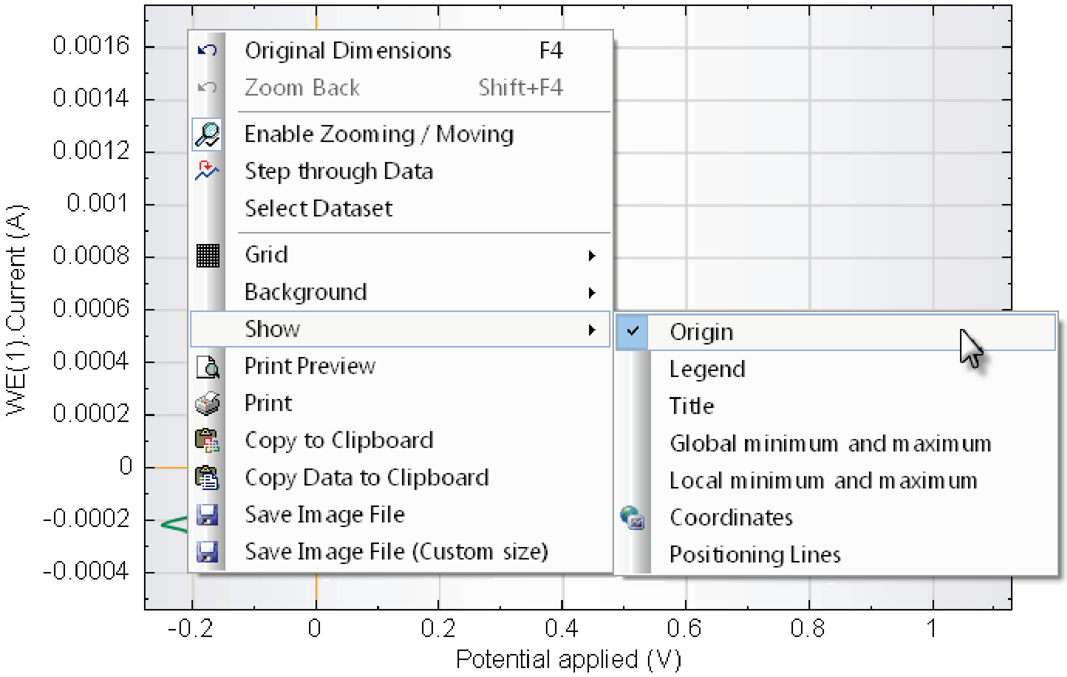


Figure 4.72 – The right-click menu and the show sub-menu

From the Show sub-menu, you can select some additions for the plot:

- Show Origin (2D)
- Show Legend (2D, 3D)
- Show Title (2D, 3D)
- Show Global minimum and maximum (2D)
- Show Local minimum and maximum (2D)
- Show Coordinates (2D)
- Show Positioning Lines (2D)

All these additions can be toggled on and off at any time and the changes can be saved in the database using the save button, .

Show Origin (2D plots only): this option will display the origin of the plot (0, 0) on the 2D graph (two orange lines are shown). The scaling of the plot is adapted if required. Figure 4.73 below shows the same cyclic voltammogram plot with its origin.

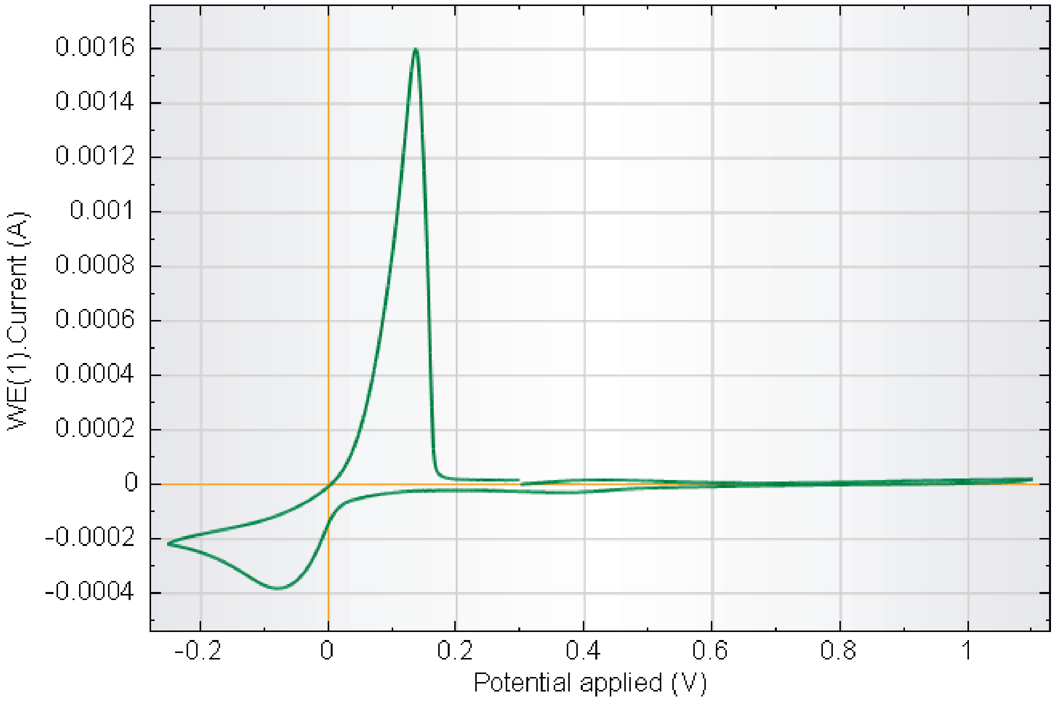


Figure 4.73 – Showing the origin of the plot

Show Legend (2D & 3D plots): this option will display a legend for the data set(s) plotted in the 2D graph, using the color and markers, as well as the name of the plot(s). Figure 4.74 shows a typical example.

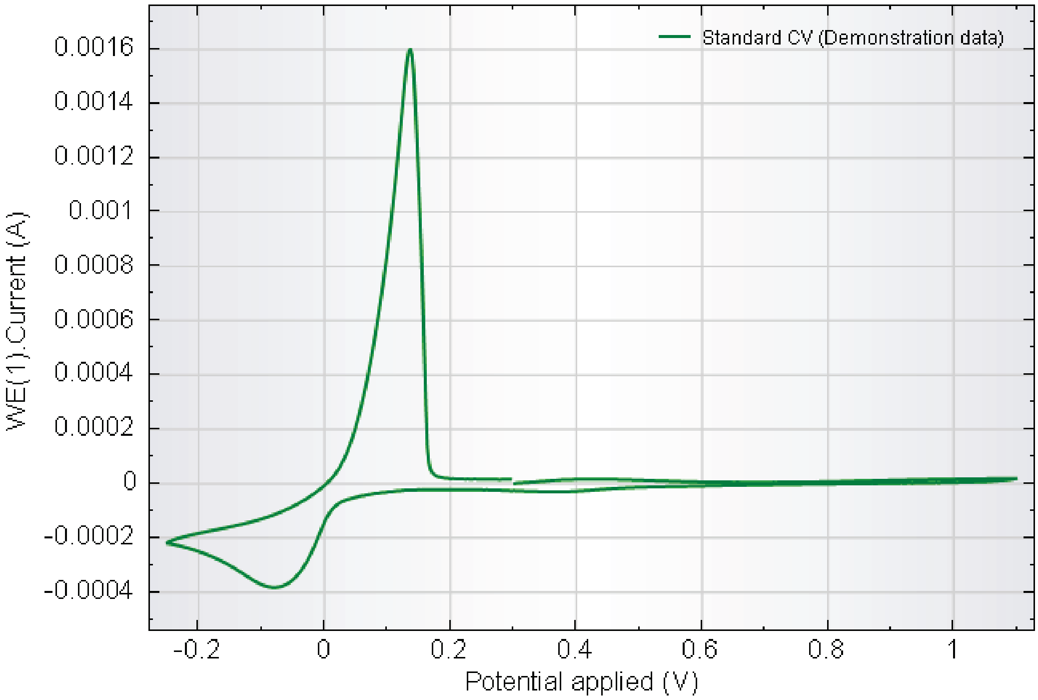


Figure 4.74 – Showing the legend of the plot

Note

The legend displays the serial number or the instrument description from the data set between brackets (see Figure 4.74). This provides a convenient way of identifying points from different data sets. If the *Instrument description* field is filled in the dataset, this will be indicated between brackets in the legend (Please refer to Section 4.1.1 for more information). If no *Instrument description* is provided, the serial number is indicated instead (see Figure 4.75).

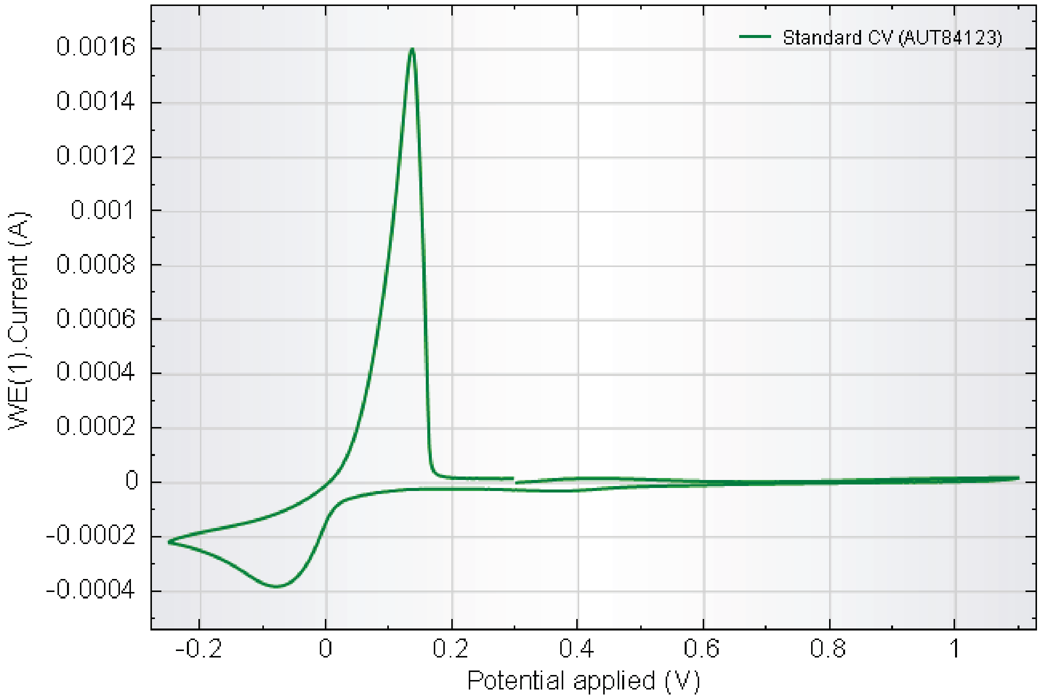


Figure 4.75 – The serial number of the instrument is provided in between brackets when no instrument description is provided

The position, the font and the style of the legend can be edited using a specific right-click menu. A total of eight positions are available for a 2D plot and four positions for a 3D plot as well as three different border styles. The legend can also be hidden from view using this menu (see Figure 4.76). The legend settings are set independently for each type of plot.

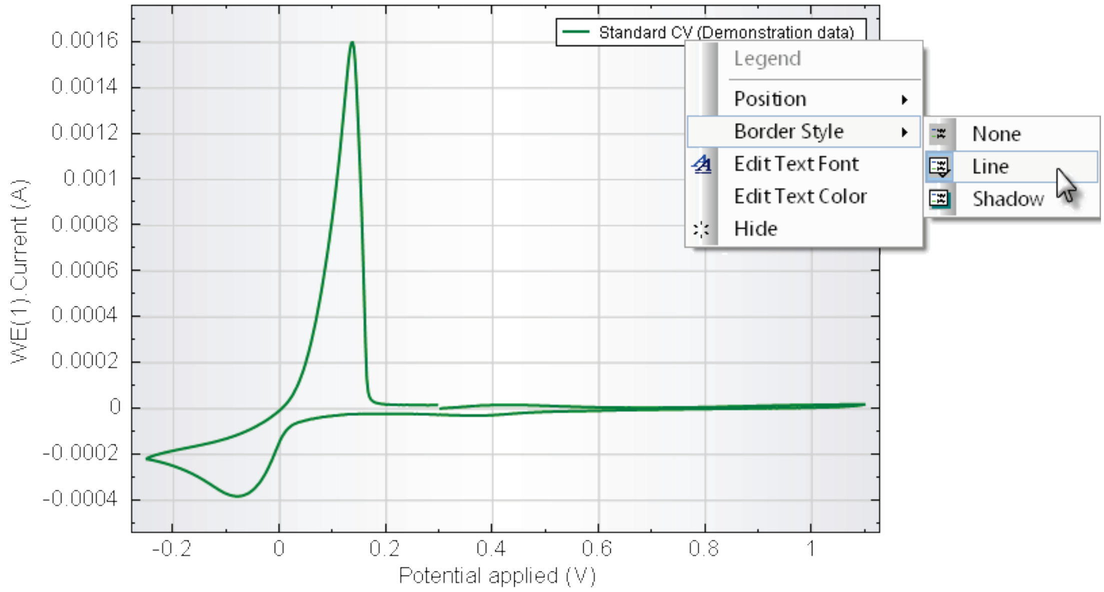


Figure 4.76 – The legend sub-menu for position, border style and font edition

Show Title (2D & 3D plots): this option will display a title for the data set(s) plotted in the 2D graph. As with the legend, the format of the title and of the subtitle can be edited using the right-click menu. Their position can be chosen from three predefined settings, both for the 2D and for the 3D plots, although the settings are independent for each type of plot. Figure 4.77 shows the default title for the 2D plot, and the right-click menu used for editing text, font and position of the title and the subtitle.

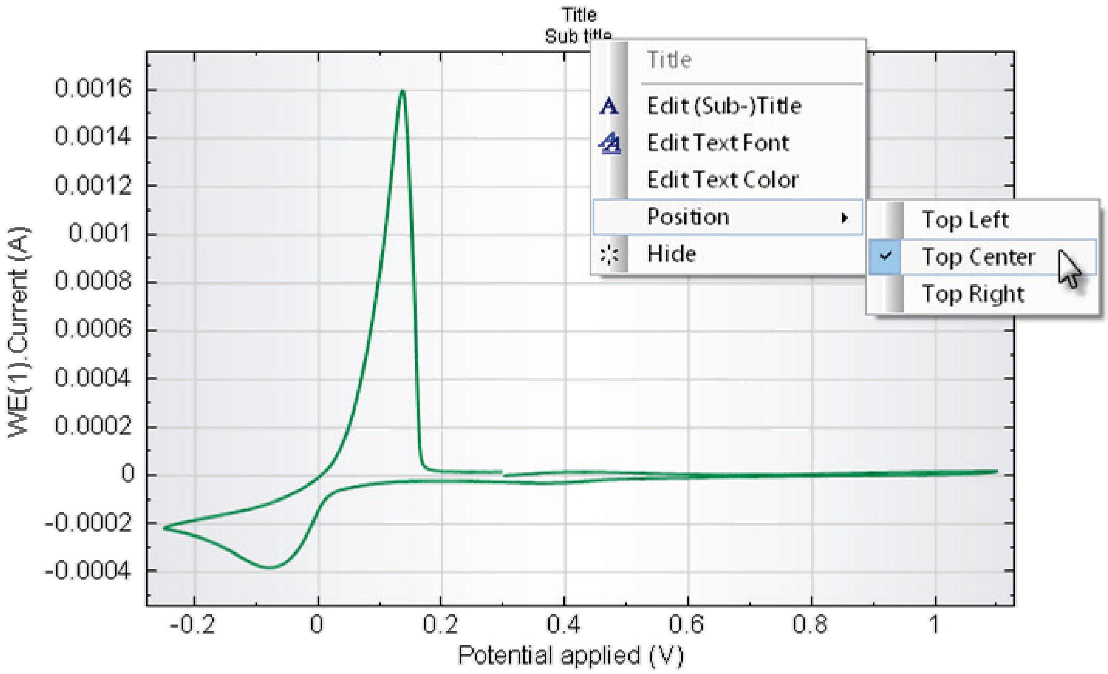


Figure 4.77 – Displaying the title and the subtitle and using the right-click menu for editing

Note

It is also possible to edit the title and the subtitle by double clicking it.

Show Global minimum and maximum (2D plots only): this option displays the points of the plot corresponding to the **absolute** lowest and the highest value of the signal plotted on the Y-axis. Figure 4.78 shows the location of the Global minimum and maximum for the cyclic voltammogram of example #1. The labels can be removed by selecting this option once again.

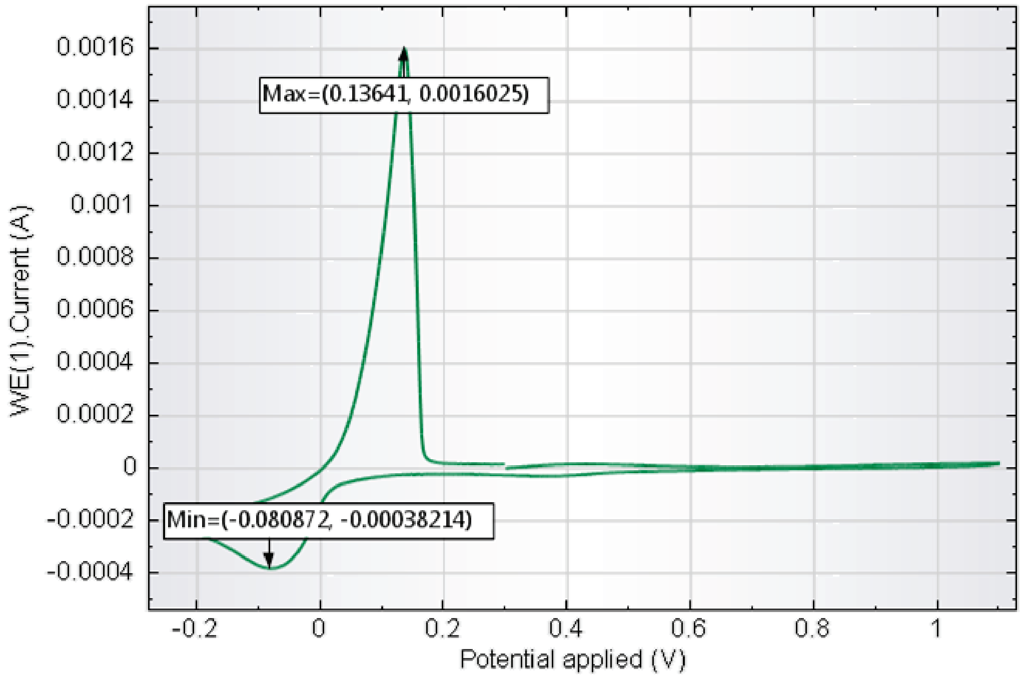


Figure 4.78 – Showing the position of the Global minimum and maximum

Note

In the case of an overlay plot using two different Y-axes, the maximum and minimum for both axes are displayed.

Show Local minimum and maximum (2D plots only): this option displays the points of the plot corresponding to the **relative** lowest and the highest value of the signal shown on the Y-axis. When the automatic scaling option is used for the Y-axis, the Local minimum and maximum correspond to the Global minimum and maximum. Figure 4.79 shows the location of the Local minimum and maximum for a specific portion of the cyclic voltammogram of example #1. The labels can be removed by selecting this option once again.

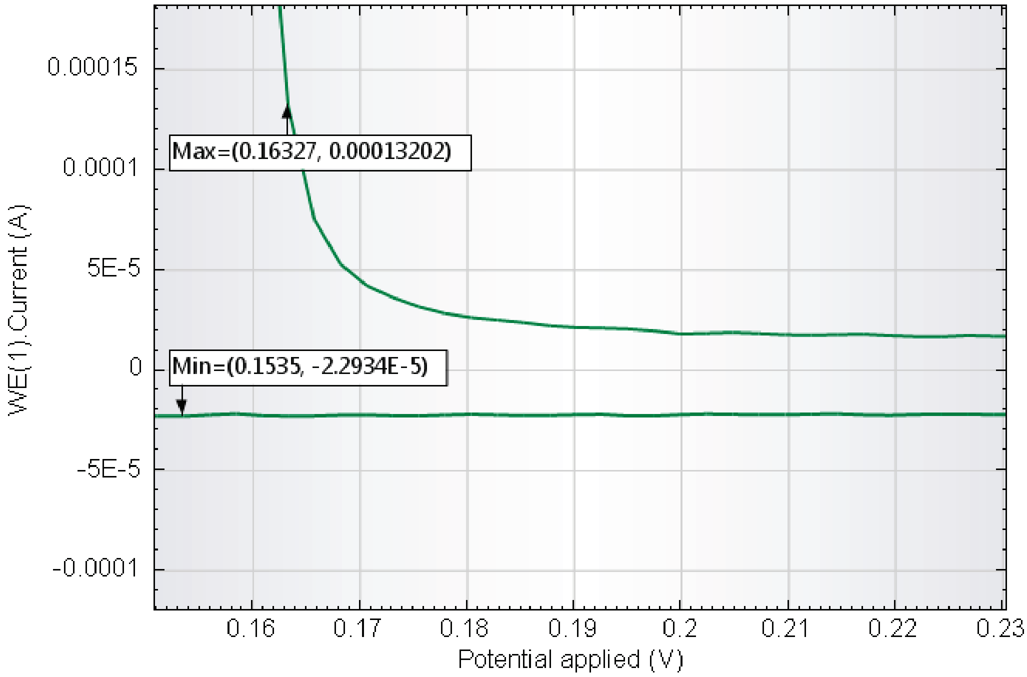


Figure 4.79 – Showing the position of the Local minimum and maximum

Note

It is not possible to show the Global and the Local minima and maxima. Selecting one of the two options automatically deselects the other one.

Show coordinates (2D plots only): this option displays the coordinates of the mouse cursor in the 2D, as a tooltip. The coordinates shown depend on the scaling and the types of axes chosen for the plot. The coordinates are refreshed when the mouse pointer is moved. Figure 4.80 shows the results of this option on the 2D plot.

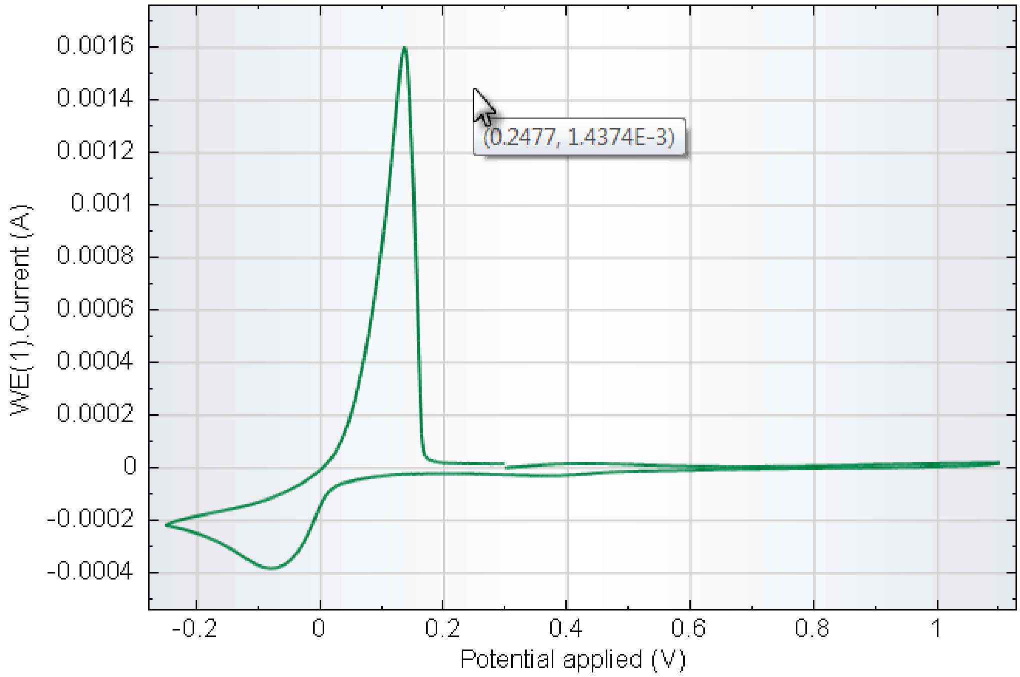


Figure 4.80 – Showing the coordinates of the mouse pointer on the plot

The displayed coordinates correspond to the position of the mouse pointer expressed in terms of the signals chosen to plot the data. In the example shown in Figure 4.80, the X-coordinate corresponds to the applied potential and the Y-coordinate corresponds to the measured current.

Note

In the case of an overlay of two or more plots, using two different Y-axes (see Section 4.4), the Show coordinates option will display two different sets of coordinates. The first set corresponds to the coordinates relative to the left (default) Y-axis. The second set corresponds to the coordinates relative to the right Y-axis (see Figure 4.81).

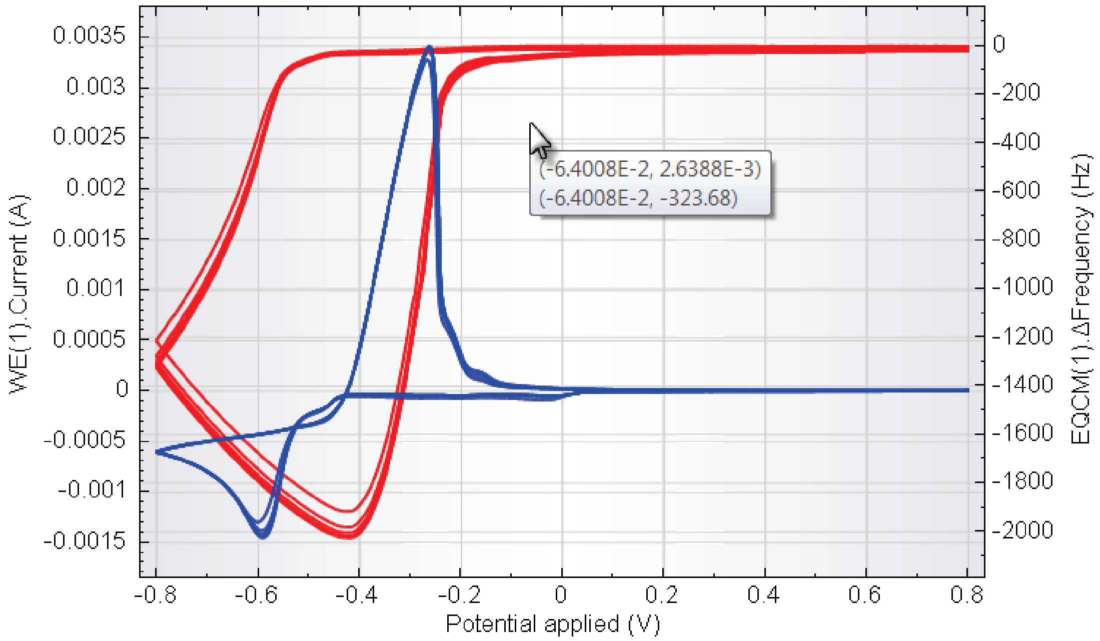


Figure 4.81 – Showing the pointer coordinates for a dual Y-axis overlay plot

Show Positioning lines (2D plots only): this option will display the two lines, following the position of the mouse pointer and helping in locating the position of the pointer on the screen (Figure 4.82).

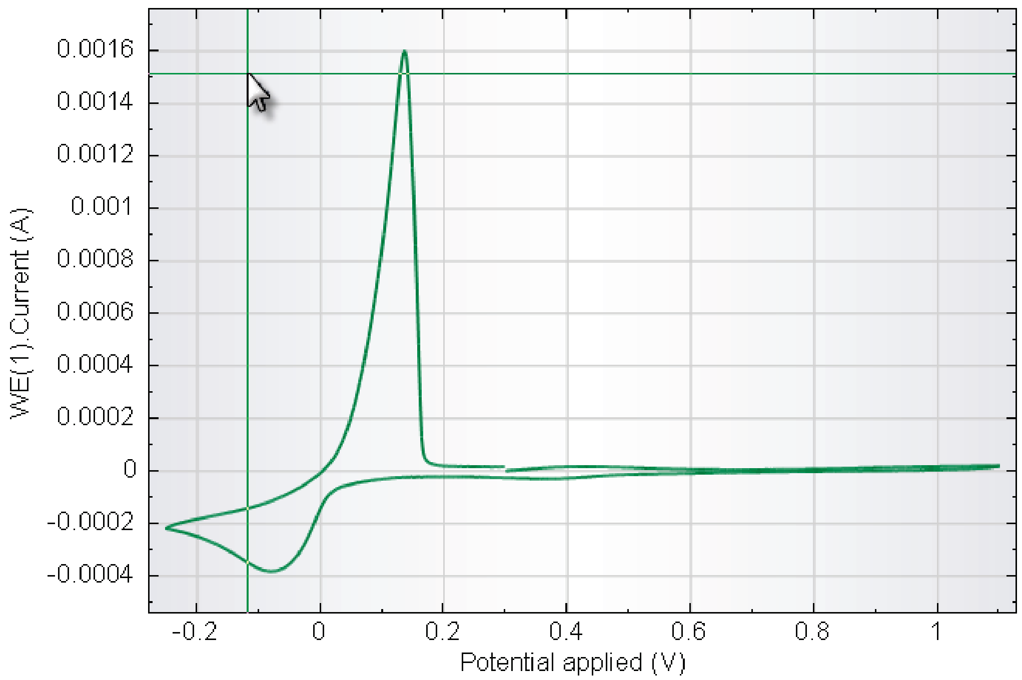


Figure 4.82 – Showing the positioning lines

4.7.4 – Stepping through data

Most of the additions that were described in the previous part of this section are general additions, which can be added to any plot, regardless of the plotted data. In some cases, more data specific additions could be required. NOVA has a convenient labeling function that can help you achieve this.

Right-click anywhere on the plot and choose the *Step through Data* option (see Figure 4.83).

Note

The *Step through data* option deactivates the *Enable Zooming / Moving* option.

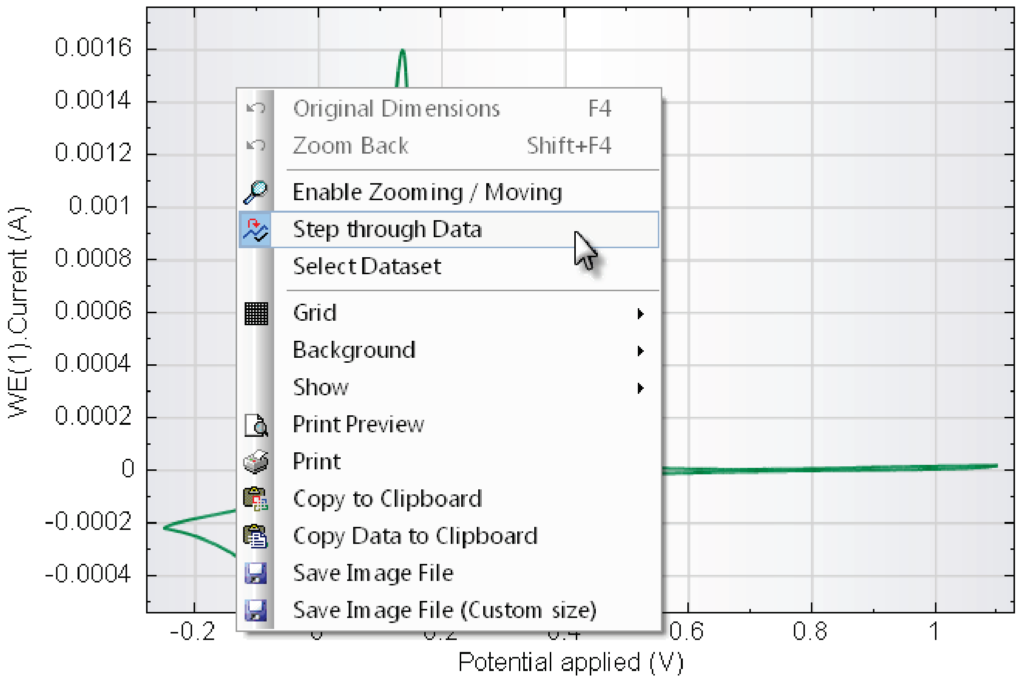


Figure 4.83 – Selecting the stepping through data option

This will change the mouse pointer to a "*hand*". Click anywhere on the displayed plot. A tooltip label will appear, together with an arrow, locating the point you just clicked and displaying some information about the point: index of the point in the data set, X, Y and Z coordinates of the plot (see Figure 4.84).

Note

The information shown in the tooltip depends on the signals used for plotting the experimental data. In the example shown in Figure 4.84, the X, Y and Z signals are Potential applied, WE(1).Current and Time, respectively.

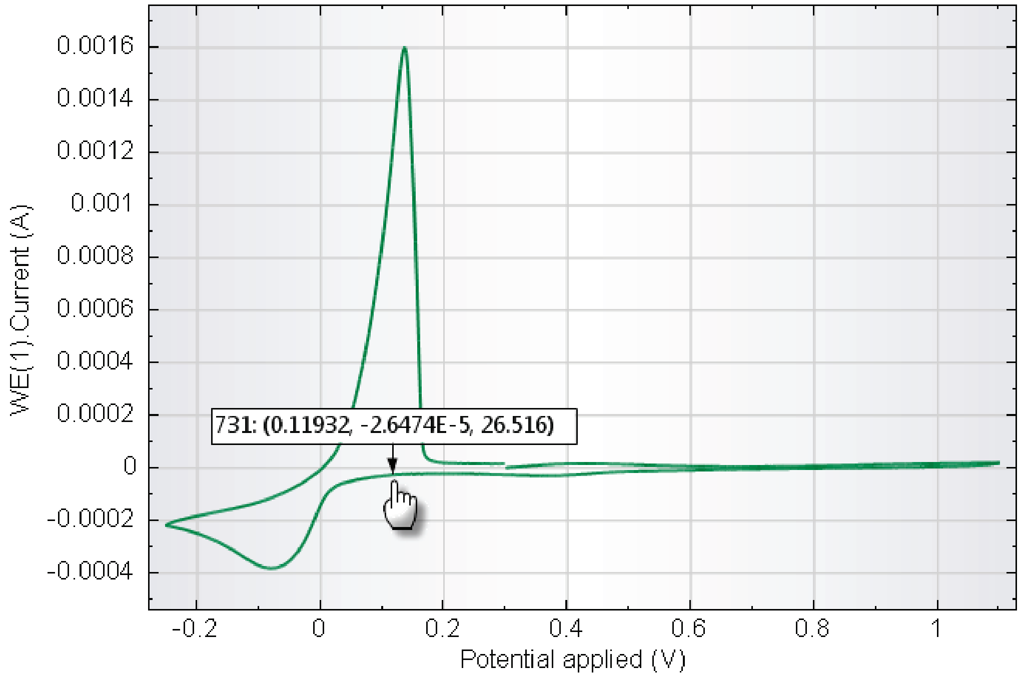


Figure 4.84 – Displaying data information using the step through data option

Clicking anywhere else on the plot will display the same information for the new point. Holding the **left** and **right** arrow keys on the keyboard will walk through the whole data set, in reverse and forward direction, respectively. It is also possible to scroll the mouse wheel up or down, if available, to walk through the data. At any time, you can press the **insert** key on the keyboard to add the label and the locating arrow to the plot (see Figure 4.85).

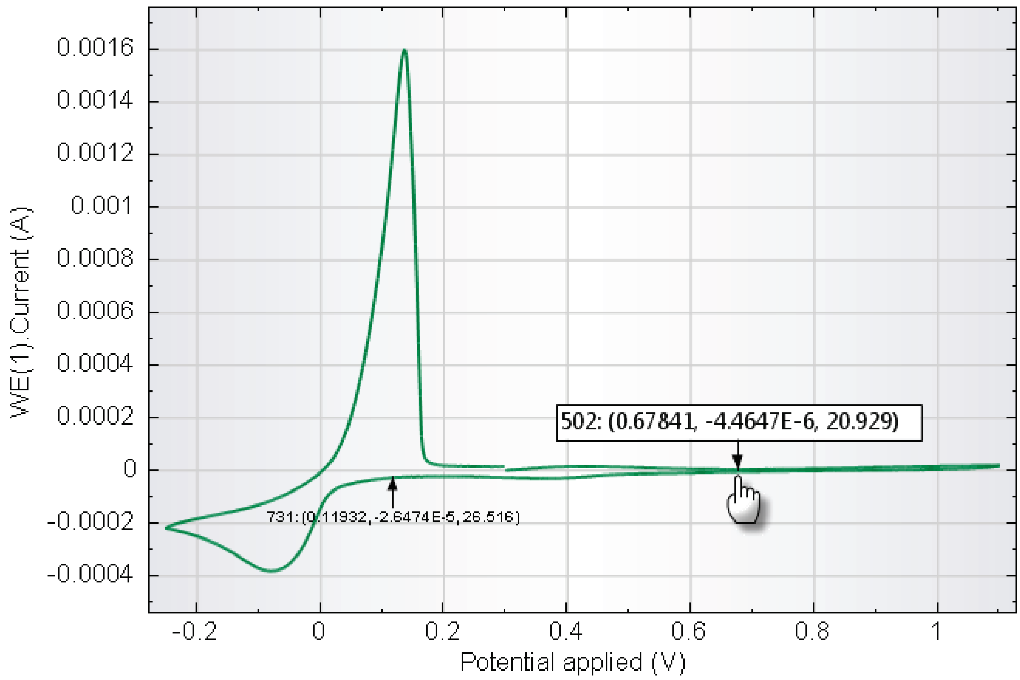


Figure 4.85 – Walking through the data using the keyboard arrows or the mouse wheel

You can add as many labels as you want to the plot using the insert key. Pressing the **home** key, automatically moves the marker to the first point of the data set and pressing the **end** key will move to the last point of the set. Pressing the **page up** and **page down** keys moves the next or the previous data set in an overlay.

Note

Holding the CTRL key while walking through the data will advance the arrow pointer using a 10 step. Holding both the CTRL and the SHIFT key will advance the arrow pointer using a 100 step.

To stop walking through the data points, click the *Step through Data* option again from the right-click menu.

You can further edit the labels that were added to the plot by right-clicking them. You can change the label text, font and direction, or remove a label, or move it to a more convenient location. Figure 4.86 shows an example of an inserted and edited label, positioned on the maximum current.

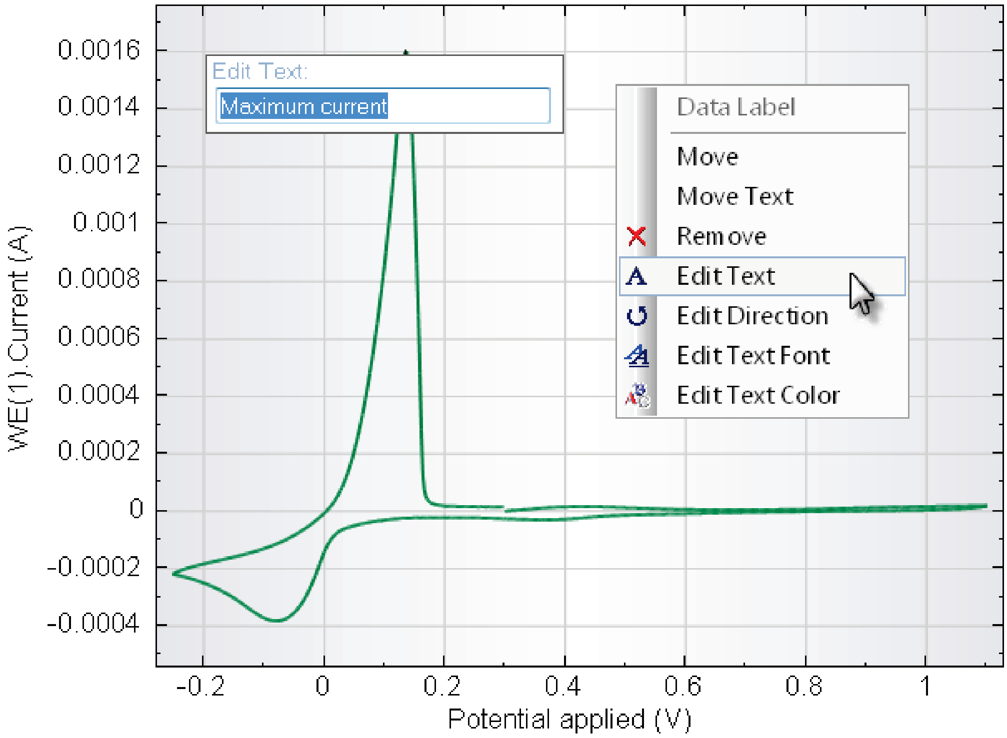


Figure 4.86 – Edited label and the edit menu

Note

Similarly to the other additions to the plots, these labels are independently placed for each type of plot. Saving the changes to the data set will also save the added labels.

4.7.5 – Axes and labels

The final part of the customization of the plot will focus on the axes and the labels. Some of the options are common to both types of plots, while other options are only available for a specific type. These options can be accessed by right-clicking one of the axes of the plot (see Figure 4.87). A description of these options is given below.

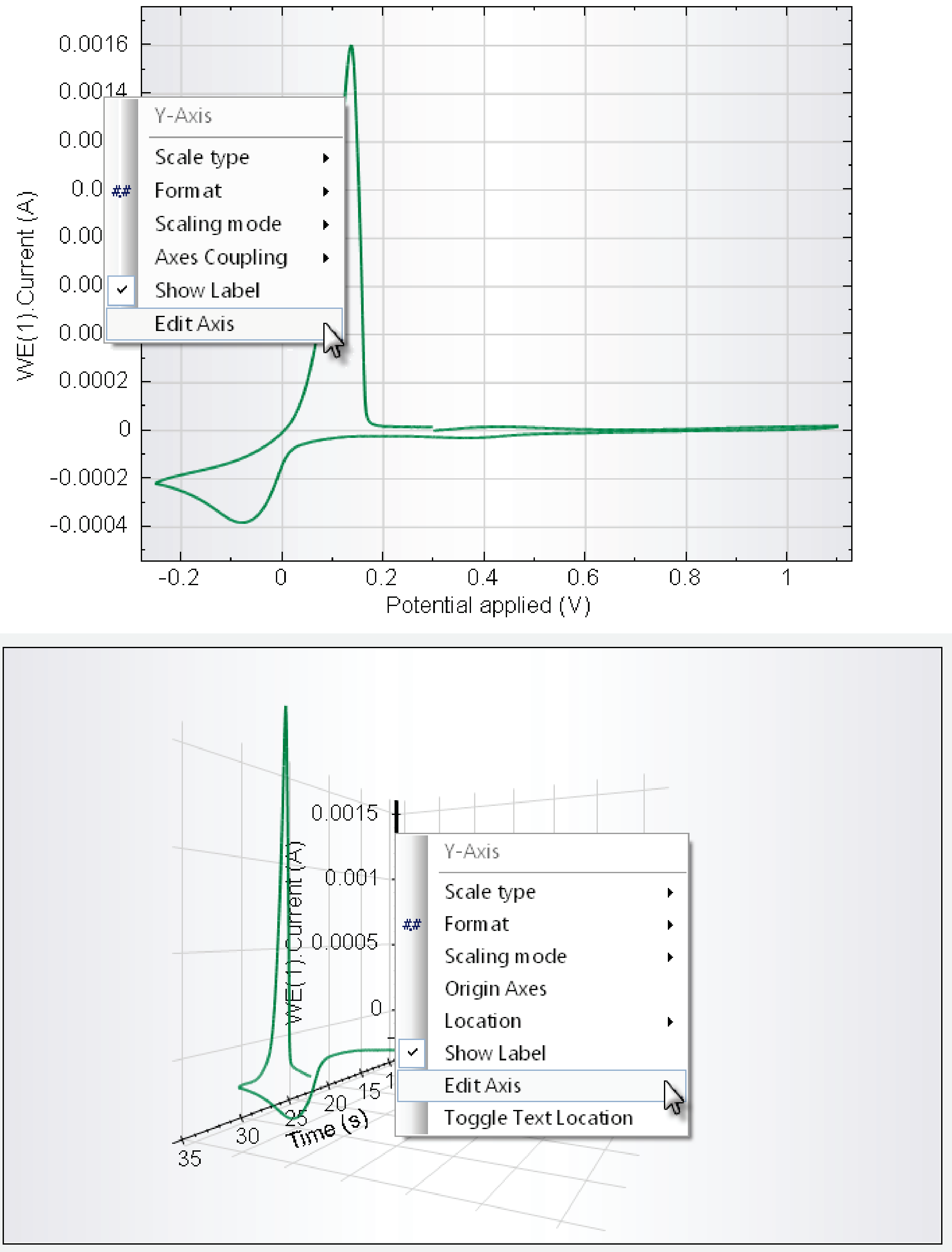


Figure 4.87 – Accessing the axes options in a 2D plot (top) and a 3D plot (bottom)

4.7.6 – Common options

- **Scale Type:** choose from linear, logarithmic absolute values, square root, π multiples and Time. The direction of the axis can also be reversed.
- **Format:** choose number formatting from general, engineering, scientific, decimal, exponential and squared values, and set the required precision.

- **Scaling mode:** choose from automatic, fixed (only for 2D plots), or fixed minimum size. **Automatic** scaling will set the X and Y scaling to its optimal setting in order to display every data point. **Fixed** scaling allows setting the scaling of an axis in such a way that only a specific part of the axis is displayed (only for 2D plots). **Fixed minimum size** scaling will plot the data using any scaling provided that displays all the data points on the screen. If some data points are outside of the chart, the scaling will be automatically adjusted.
- **Show Label:** toggle the labels on/off.
- **Edit Axis:** displays the Edit Axis window through which the settings used for the axis can be modified (see Figure 4.88).

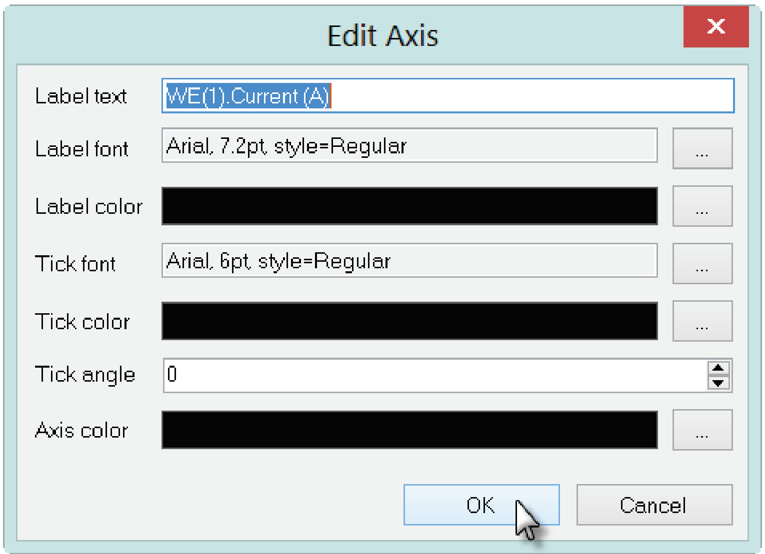


Figure 4.88 – The Edit Axis window can be used

Note

The **Tick Angle** setting is not available in 3D plots. For 2D plots, the tick angle can be set to any value between 0 to 90 degrees.

4.7.7 – 2D Specific options

- **Axes Coupling:** choose from independent, iso-metric, or iso-tropic.

4.7.8 – 3D Specific options

- **Origin Axes:** overrides any axis position setting by moving the intersection of the axis at the origin of the plot (point 0, 0, 0).
- **Location:** set the position of the axis relative to the scale of the other axes. The default setting uses the minimum of each axis to locate the origin of the 3D plot. Choose maximum to locate the axis at the highest value of the first available axis. Choose maximum alternate to locate the axis at the

highest value of the other axis (example: choosing the maximum location for the X-axis will move the axis to the highest value of the Y-scale while choosing the maximum alternate location for the X-axis will move the axis to the highest value of the Z-axis. The same options for the Y-axis will move the axis to the highest value of the X-axis and the Z-axis, respectively).

- **Toggle Text Location:** changes the position of the axis labels and tick labels relative to the axis.

Using these advanced features, a given data set can be plotted in a very clear way and the plot can then be pasted into a document. When editing is finished, the plot can be exported to another application, either directly through the clipboard or to an image file on the hard drive.

From the right-click menu, choose the **Copy to Clipboard** to copy the whole plot (2D or 3D) or select the Save **Image File** option to save the plot as a picture on the hard drive. A number of file types are available (BMP, TIF, WMF, PNG, GIF, JPG). The Portable Network Graphics (PNG), which offers a lossless compression of the image, is the default file format used in NOVA. Alternatively, the plots can be printed using the **Print** command in the right-click menu (a print preview is also available).

4.7.9 – Zooming in and out

Zooming in on some specific areas of the plot is a convenient way to get more insight on the measured data. NOVA allows users to zoom in and out in several ways, using the mouse buttons and, if available, the mouse wheel. The zoom functions are available if the *Enable Zooming / Moving option* is active (right-click – Enable Zooming/Moving).

4.7.10 – 2D plots – Zooming (Box mode)

Zooming in on specific area of a 2D plot can simply be done by dragging a box around the area of interest. Left click on a spot of the plot and while holding the left mouse button pressed, move the mouse in a direction to draw a box around the area of interest (see Figure 4.89). Release the mouse button to complete the operation.

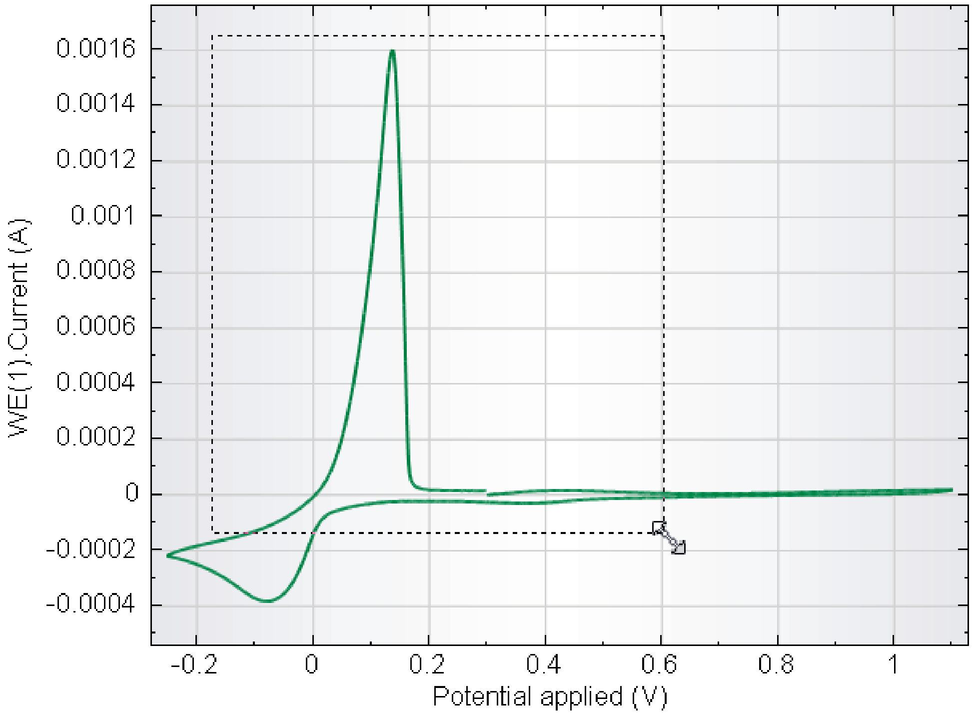


Figure 4.89 – Zooming in by dragging a box around the area of interest

It is possible to return to the original plot settings by choosing the **Original Dimensions** instruction from the right-click menu or by pressing the shortcut **F4** key on the keyboard. If several boxes have been drawn, it is possible to return step-by-step to the initial plot by choosing the **Zoom Back** option from the same menu or by using the shortcut **Shift + F4** key combination (see Figure 4.90).

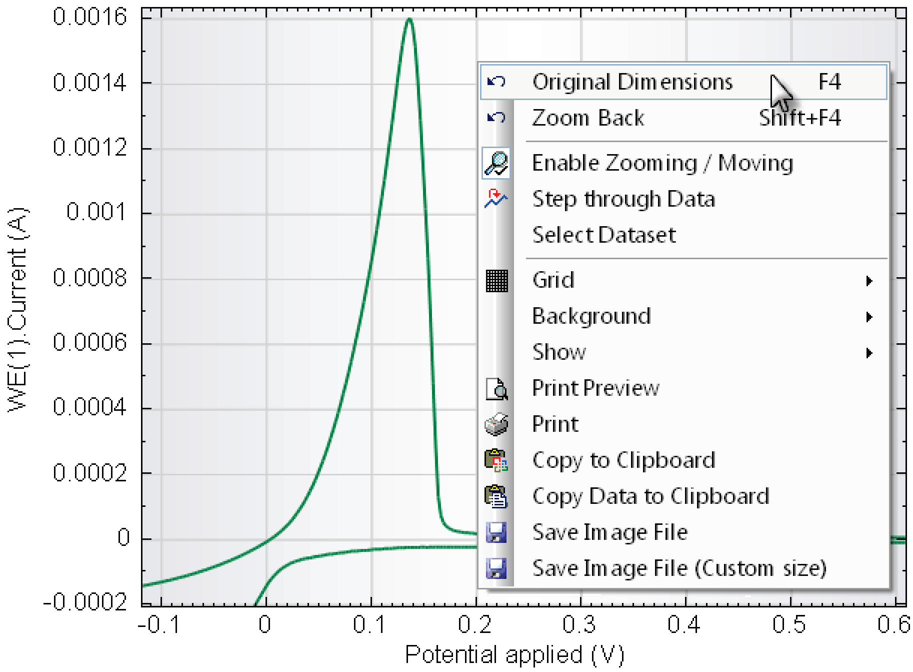


Figure 4.90 – Enabling zooming and zooming back using the right-click menu

Note

It is only possible to zoom on the data if the *Enable Zooming / Moving* option is selected. The orange background and the check mark in the right-click menu will indicate which option is currently active (see Figure 4.91).

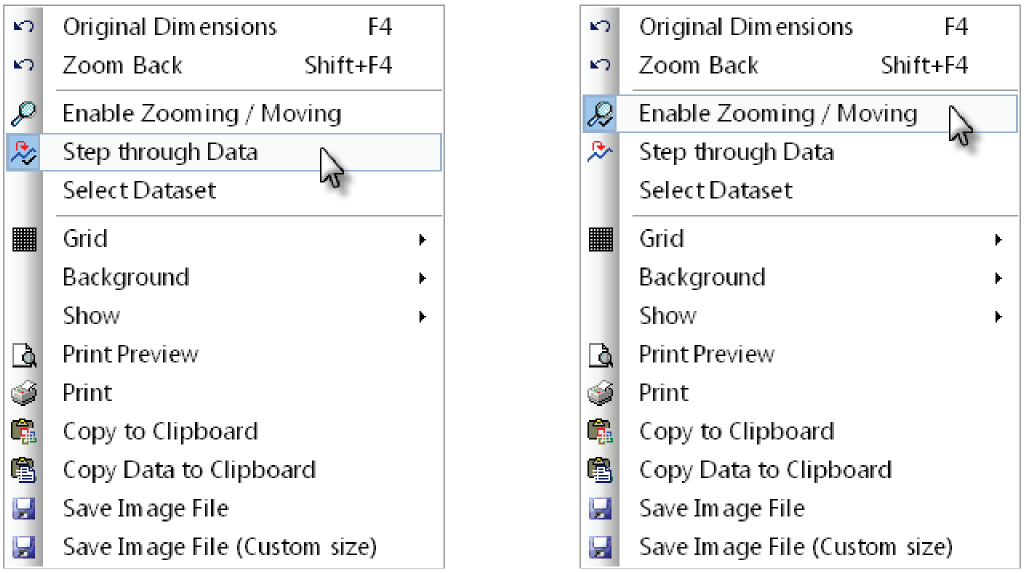


Figure 4.91 – Switching from Step through data to Enable zooming/moving

4.7.11 – 2D Plots – Zooming (Scroll mode)

As an alternative to dragging a box around the area of interest of the plot, the mouse wheel can be scrolled forwards and backwards to zoom in and out on the plot. This option will always keep the centre of the plot in the same position as the wheel is scrolled in either direction (see Figure 4.92).

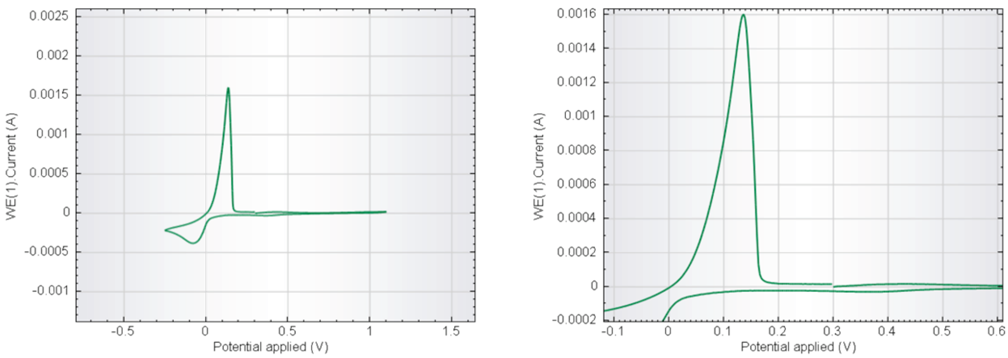


Figure 4.92 – Scroll zoom. Scrolling in (left) and scrolling out (right)

Note

It is possible to zoom in and out by using the + and – keys, respectively. Using the CTRL key in combination with the + and – keys, it is possible to zoom in and out faster, by a factor 10. Using both the SHIFT and the CTRL key, the rate increases by a factor 100.

4.7.12 – 2D Plots – Zooming (Sliding mode)

A third way of zooming on the data is available by clicking and sliding the mouse left/right for the X-axis and up/down for the Y-axis to expand or contract the scaling of the axes. The clicked position on the axis will stay in the same position while sliding the axis (see Figure 4.93).

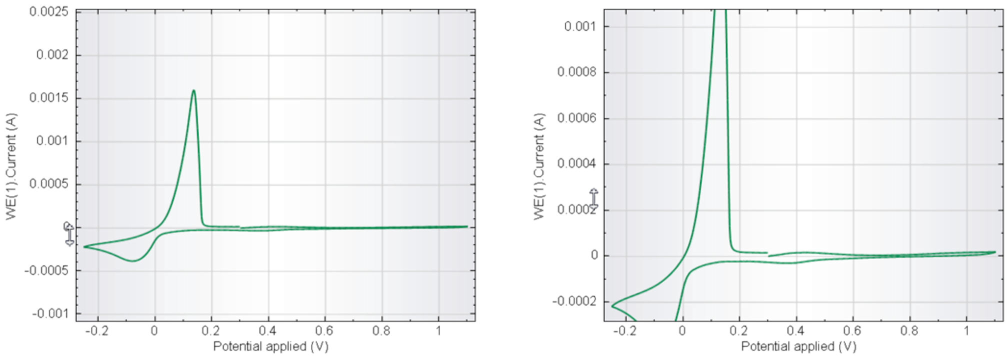


Figure 4.93 – Slide zoom: expanding the Y-axis (left) and contracting it (right) using the sliding zoom function

4.7.13 – 2D Plots – Moving (Dragging mode)

The 2D plot can be moved around using the wheel mouse button (if available). This option is particularly useful to move hidden parts of the plot into view after zooming in on the plot. To use this feature, click and hold the wheel mouse button and drag the hidden part of the plot into view. While the wheel mouse button is pressed, the mouse pointer changes from an arrow to a cross-like pointer. Figure 4.94 illustrates this option.

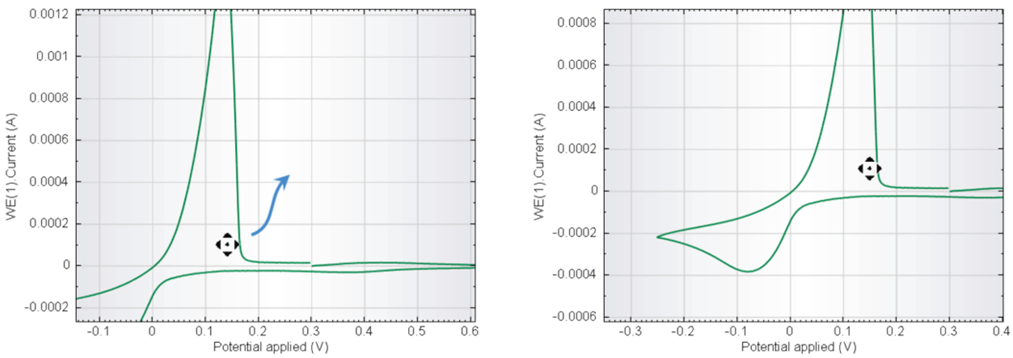


Figure 4.94 – Using the moving feature

Note

Using the keyboard, it is possible to move the plot relative up, down, left and right by using the corresponding keys. In combination with the CTRL key and the SHIFT key, the movement can be accelerated.

4.7.14 – 3D – Zooming (Scrolling mode)

The zooming functionalities for a 3D plot are limited to scrolling in and out using the mouse wheel. Depending on the location of the mouse pointer, the zooming function will affect the whole plot or a specific axis. If the mouse pointer is located anywhere on the plot except on one of the axes, scrolling the mouse wheel up and down zooms in and out on the plot (see Figure 4.95).

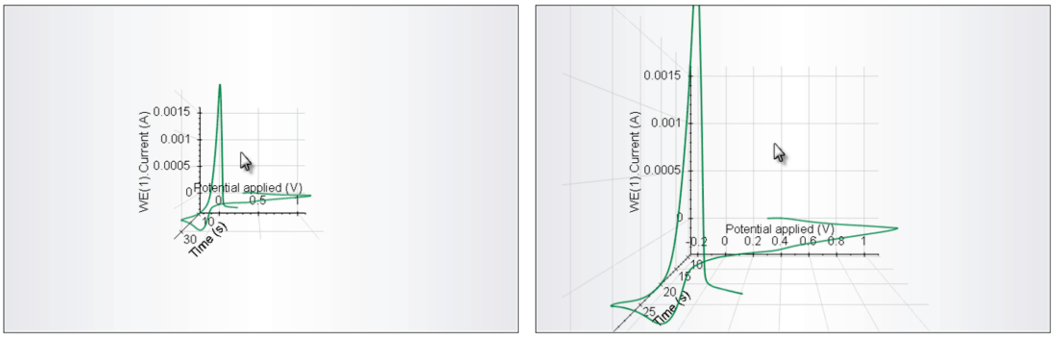


Figure 4.95 – Using the scroll function to zoom in on the 3D plot

If the mouse scroll function is used while the mouse pointer is located on top of one of the 3 axes of the plot, the axis will expand or contract depending on the scrolling direction (see Figure 4.96).

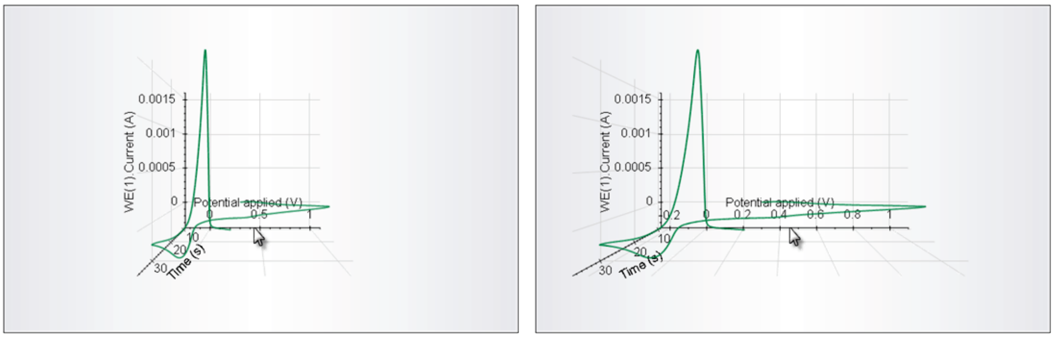


Figure 4.96 – Expanding the X-axis using the mouse scroll function

Note

Since 3D plots can be displayed in many different ways, it might be convenient to set one specific view of the plot as a *User-defined view*. To do this, right-click on the 3D plot, and from the Dimensions menu, choose the Set as User Dimensions option (or use the Shift + F12 shortcut). After defining the user dimensions, you can return to this view at any time by pressing the F12 key. Hitting the F4 key will return the 3D plot to the original dimensions.

4.8 – Data analysis

The Analysis view is where the data analysis takes place. So far, this chapter has illustrated the mechanics of plotting measured data in the data analysis view, however no data analysis tools have been explored as of yet. This section will cover the available data analysis tools.

A detailed list of available analysis tools is provided in the Command list (Help menu – Tutorials – Command list).

Two type of analysis tools can be added to data sets in NOVA:

- **X/Y analysis tools:** these are analysis tools that can be directly added to a plot in the analysis view. These tools use the plotted X and Y values for data analysis. The result of these analysis tools depend on the input X and Y displayed in the plot.
- **Data set analysis tools:** these are analysis tools that can be added to one or more data sets. All the values of the data set(s) are used in the analysis.

4.8.1 – X/Y analysis tools

The X/Y analysis tools can be added to any plot in the analysis view, using the right-click menu (see Figure 4.97). This section provides an overview of the available analysis commands in this category.

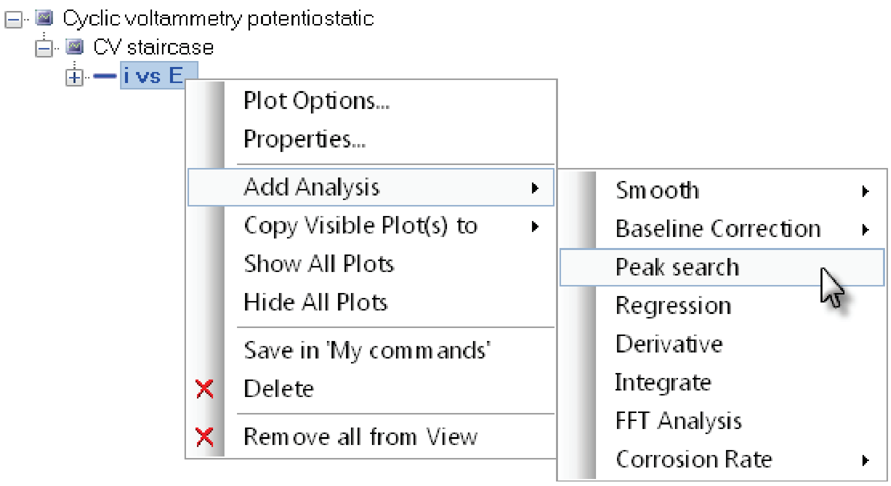


Figure 4.97 – X/Y analysis tools can be added directly to a plot in the analysis view

It is also possible to add this type of analysis by clicking the plot directly and selecting the required analysis tool using the quick access toolbar (see Figure 4.98).

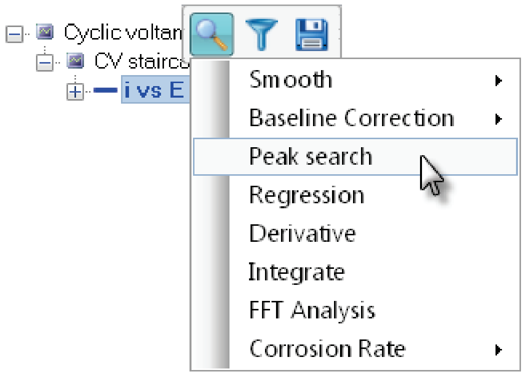


Figure 4.98 – X/Y analysis tools can be added directly to a plot using the quick access toolbar

The analysis tools in this group use the X and Y values of the plot they are added to.

4.8.1.1 – SG Smooth

The Savitzky-Golay (SG) smoothing tool can be used to remove noise from measurements in order to enhance the signal to noise ratio of experimental data.

The SG Smooth method is described in Anal. Chem.,36,1627 (1964). It involves a polynomial fit through the experimental data. This method is also called weighted moving averaging. Before the smooth routine of Savitzky and Golay is applied to the data set, spikes in the set of data can be removed.

The SG smooth method can be used with different levels of smoothing. High levels lead to heavy filtering. In NOVA, the following levels can be used:

- **Level 1:** 5-point weighed moving average
- **Level 2:** 9-point weighed moving average
- **Level 3:** 15-point weighed moving average
- **Level 4:** 23-point weighed moving average
- **User defined:** this setting can be used to customize the smoothing

The applicable smooth level depends on the number of points of the data set. The more data points, the higher the smooth level can be without modifying the curve too much.

Double click on the demo file *Demo 15 – UME LSV* entry of the demo database to load it into the data explorer frame. This file contains data points recorded in NaOH 0.2 M, in the presence of 0.05 M $[Fe(CN)_6]^{4-/3-}$ using a Pt ultra micro electrode (5 µm).

Click the i vs E plot in the data explorer frame to display the data points from this demo file (see Figure 4.99). As frequently seen with this type of measurement, the current levels are very small and affected by a significant amount of noise.

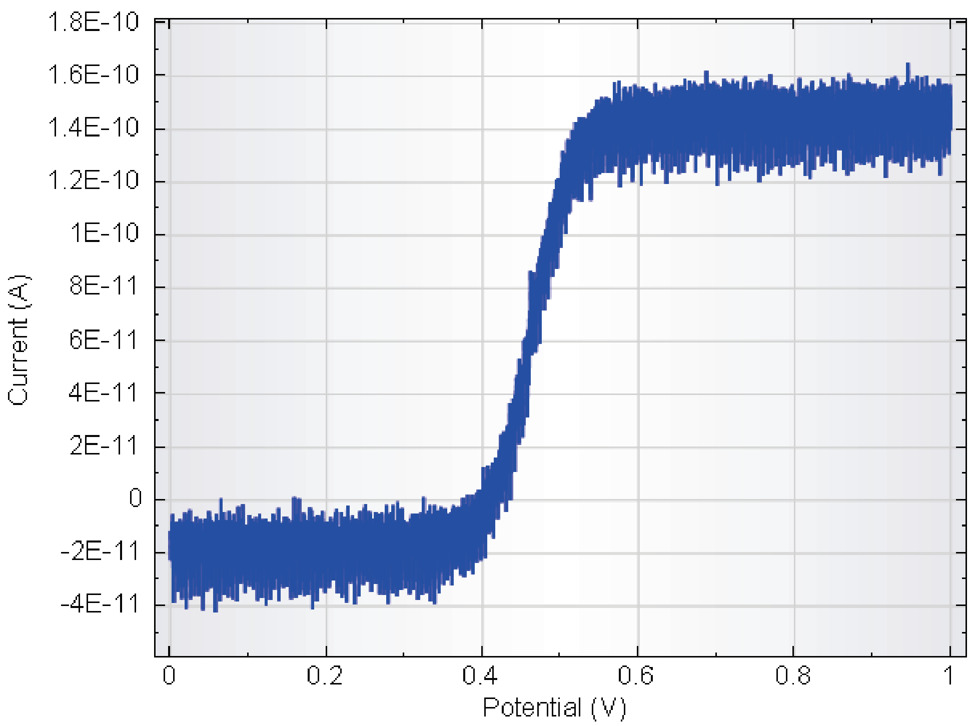


Figure 4.99 – The data from the Demo 15 data file

To use the SG Smooth tool, right-click the i vs E plot in the data explorer frame and, from the context menu, select the SG Smooth tool (see Figure 4.100).

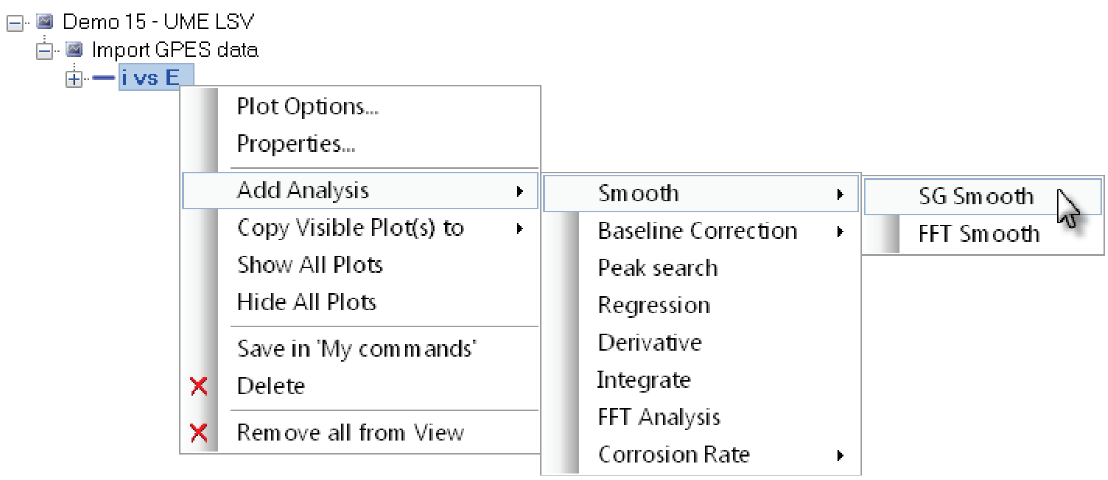


Figure 4.100 – Adding the SG Smooth tool to the data

A new item, called SG Smooth, will be added to the data set in the data explorer frame, below the i vs E plot (see Figure 4.101).

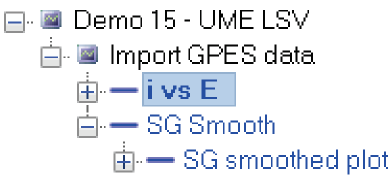


Figure 4.101 – The newly added SG Smooth tool

Click the SG Smooth item. A new area will be displayed on the right-hand side of the plot. This part of the view allows the definition of settings used in the SG Smooth tool (see Figure 4.102).

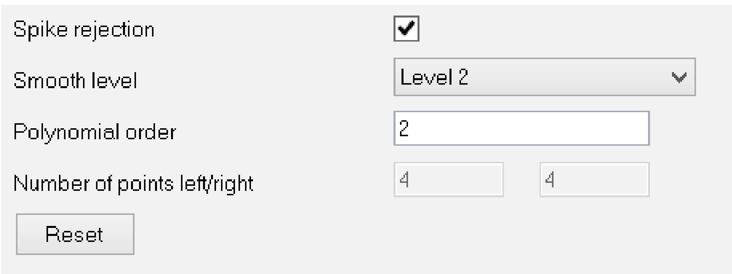


Figure 4.102 – The SG Smooth options

The following settings for the SG Smooth tool are available:

- **Spike rejection**: yes/no.
- **Smooth level:** defines the number of points in the weighted moving average function. The higher the level, the heavier the smoothing (default: Level 2).

- **Polynomial order:** defines the order of the polynomial function fitted through the data. Small order leads to heavy smoothing (default: 2).
- **Minimum peak width:** defines the minimum width of the peak, at half height, in X-axis units (0 means that this selection criterion is not used).
- **Number of points left/right:** this parameter defines the number of data points in the weighted moving average. Both values are fixed for the pre-defined Smooth levels. When the User-defined level of Smoothing is used, the number of points left/right can be defined. The larger the values, the heavier the smoothing. These two values should always be identical.

Clicking the SG Smooth item displays a preview of the smoothed data in the analysis view (see Figure 4.103).

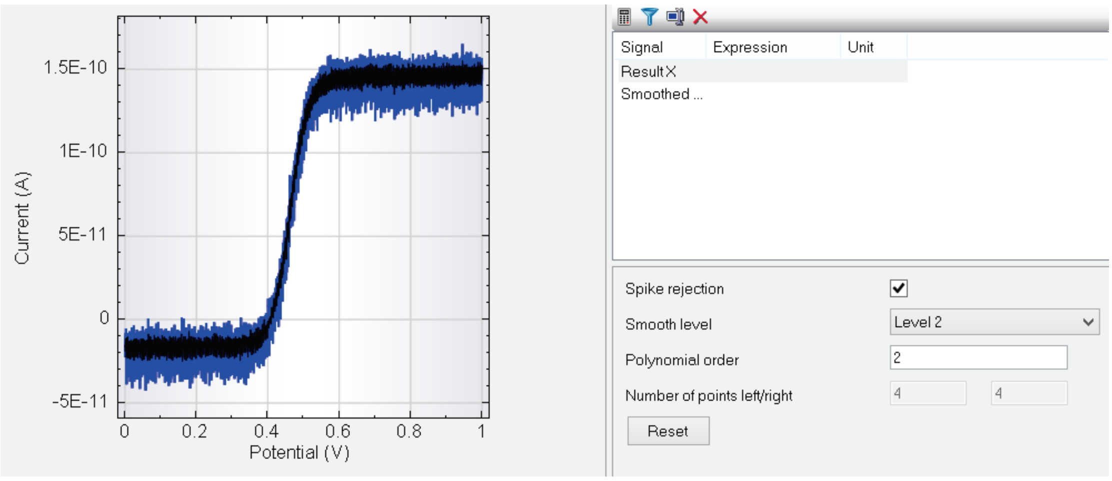


Figure 4.103 – The preview of the smoothed curve is displayed in black

Changing on the SG Smooth tool settings will automatically trigger the recalculation of the smoothing on the original data set. For example, changing the Smooth level from Level 2 to Level 4 will immediately display the same curve, with a heavier smoothing (see Figure 4.104).

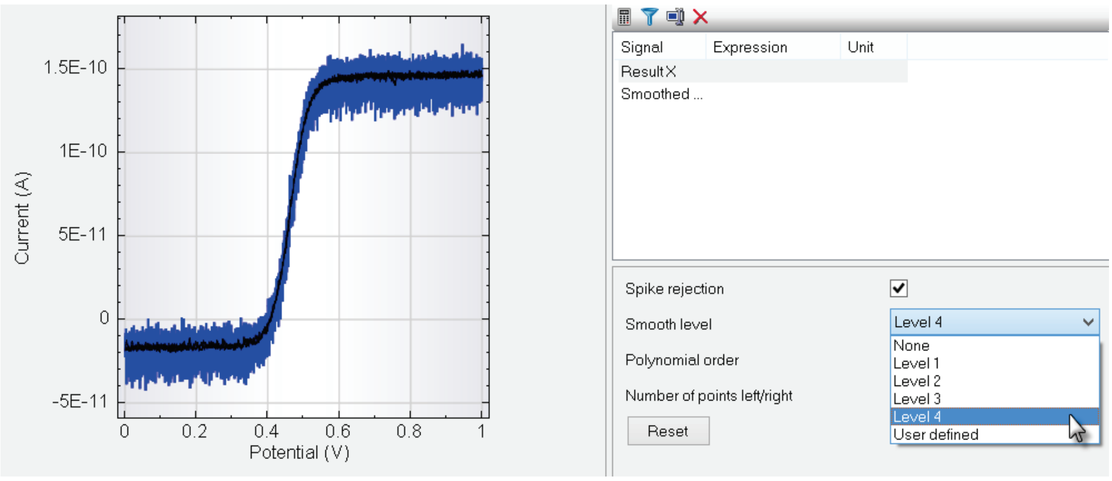


Figure 4.104 – A higher level results in heavier smoothing

The SG Smooth tool automatically generates an additional plot, called SG smoothed plot, corresponding to the result of the defined Savitzky-Golay smoothing (see Figure 4.105).

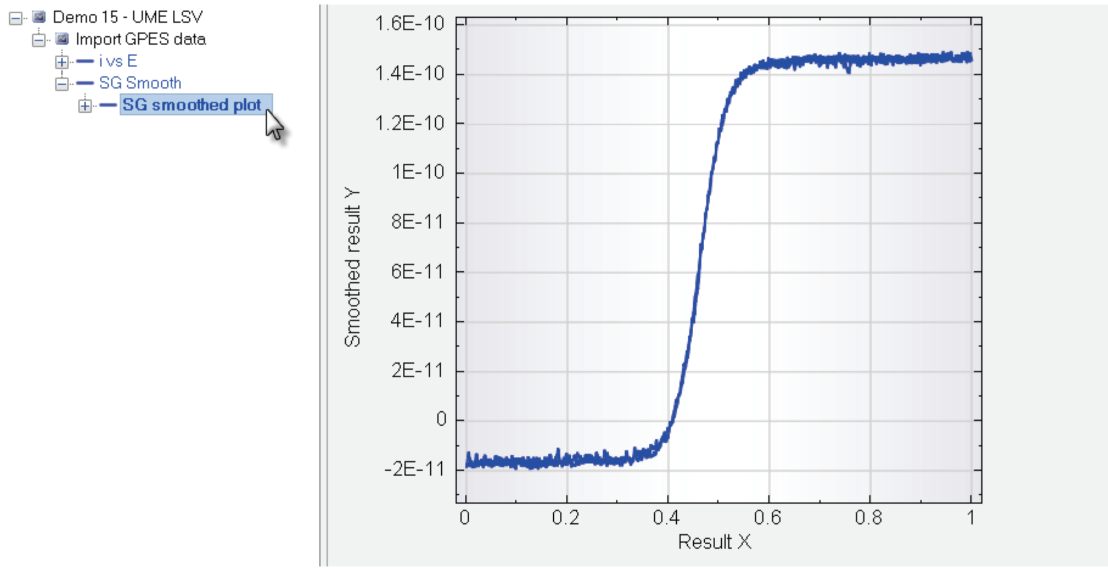


Figure 4.105 – The SG Smooth tool automatically generated a Smoothed plot

> **Note**
>
> Changing the settings of the SG Smooth tool automatically updates the Smoothed plot.

4.8.1.2 – FFT Smoothing

The FFT smoothing technique can be used to remove a specific frequency or frequency range from a data set, in order to attenuate the influence of the selected frequency or frequency range and improve the signal-to-noise ratio.

In order to identify the different frequency components present in a given data set, the FFT smooth function first transposes the source data from the time domain into the frequency domain by Fourier transformation.

The FFT smooth method can be used with four different filtering strategies:

- **Low pass:** all the contributions from frequencies higher than the user-selected cutoff frequency are rejected. This method can be used to remove high frequency noise from a measurement.
- **High pass:** all the contributions from frequencies lower than the user-selected cutoff frequency are rejected. This method can be used to remove low frequency noise from a measurement.

- **Band pass:** only the contributions from frequencies within a user-defined frequency range are kept. All frequencies that fall outside of the user defined range are rejected.
- **Band stop:** all the contributions from frequencies within a user-defined frequency range are rejected. Only the frequencies that fall outside of the user defined range are kept.

The type of FFT smoothing used depends on the noise characteristics in the measurement.

Double click on the demo file *Demo 15 – UME LSV* entry of the demo database to load it into the data explorer frame. This file contains data points recorded in NaOH 0.2 M, in the presence of 0.05 M $[Fe(CN)_6]^{4-/3-}$ using a Pt ultra micro electrode (5 μm).

Click the i vs E plot in the data explorer frame to display the data points from this demo file (see Figure 4.106). As frequently seen with this type of measurement, the current levels are very small and affected by a significant amount of high frequency noise.

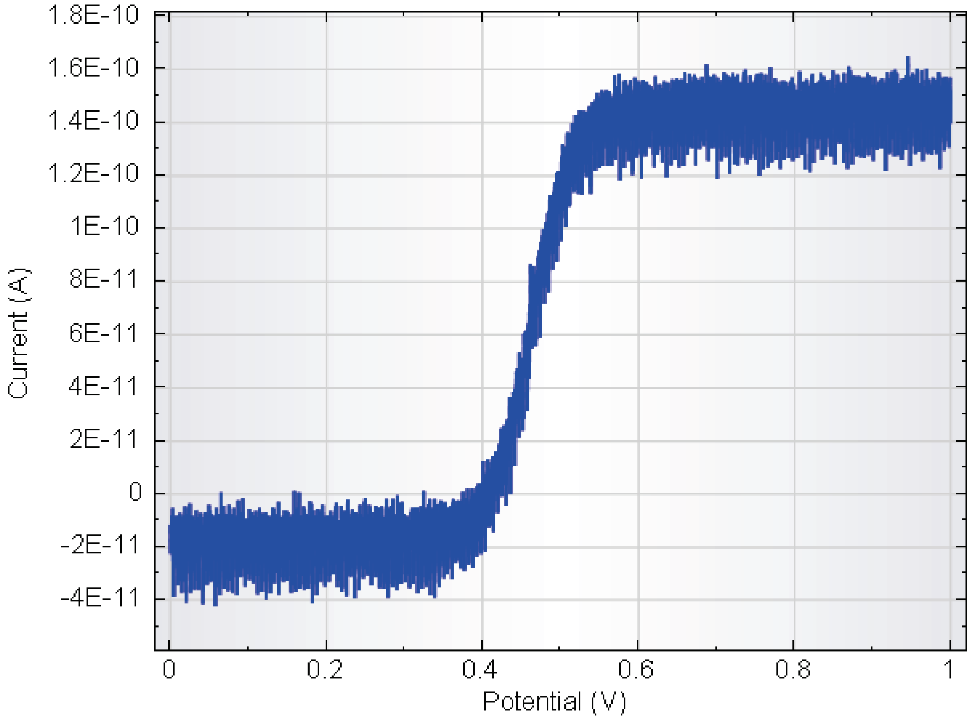


Figure 4.106 – The data from the Demo 15 data file

To use the FFT Smooth tool, right-click the i vs E plot in the data explorer frame and, from the context menu, select the FFT Smooth tool (see Figure 4.107).

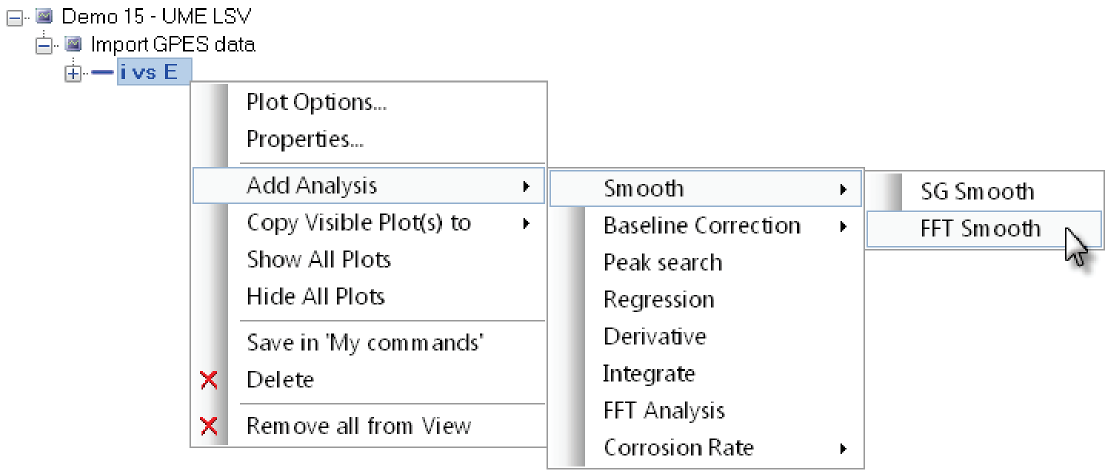


Figure 4.107 - Adding the FFT Smooth tool to the data

A new item, called FFT Smooth, will be added to the data set in the data explorer frame, below the i vs E plot (see Figure 4.108).

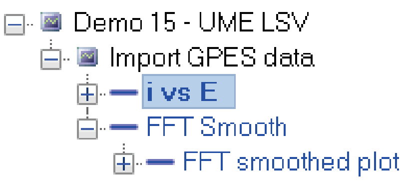


Figure 4.108 – The newly added FFT Smooth tool

Click the FFT Smooth item. A frequency domain plot will be shown. On the X axis, the frequency contributions are shown, in Hz. On the Y axis, the intensity is shown, in arbitrary units (see Figure 4.109).

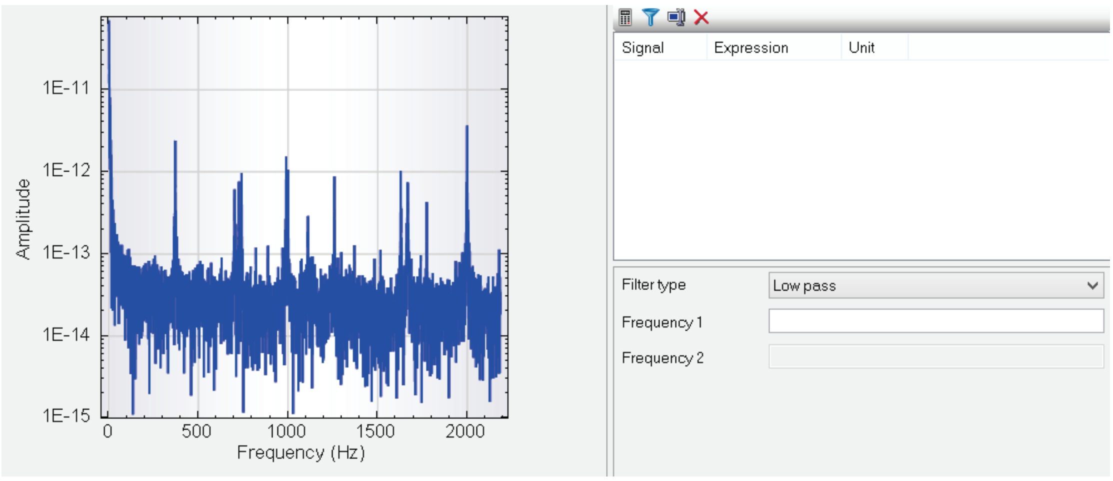


Figure 4.109 – The frequency domain plot showing the amplitude vs frequency

A new area will be displayed on the right-hand side of the plot. This part of the view allows the definition of settings used in the FFT Smooth tool (see Figure 4.110).

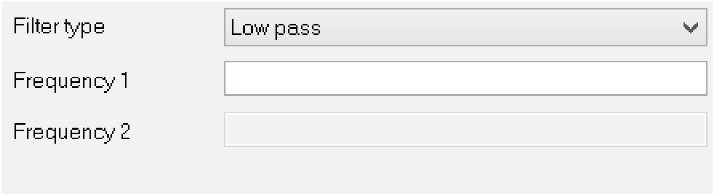


Figure 4.110 – The FFT Smooth options

4.8.1.2.1 – Low pass and high pass filtering

Using the Filter type drop-down box, one of the four filter types can be selected. The default is the Low pass filter. For the low and high pass filters, a single cutoff frequency can be manually defined in the Frequency 1 field.

Alternatively, the mouse pointer can be used to select the cutoff frequency in case of the high or low pass filter. Click the frequency domain plot at the required frequency to specify the cutoff frequency. A line will be drawn on the plot to indicate the position of the frequency in the plot (see Figure 4.111). This frequency value will also be shown in the FFT Smooth options.

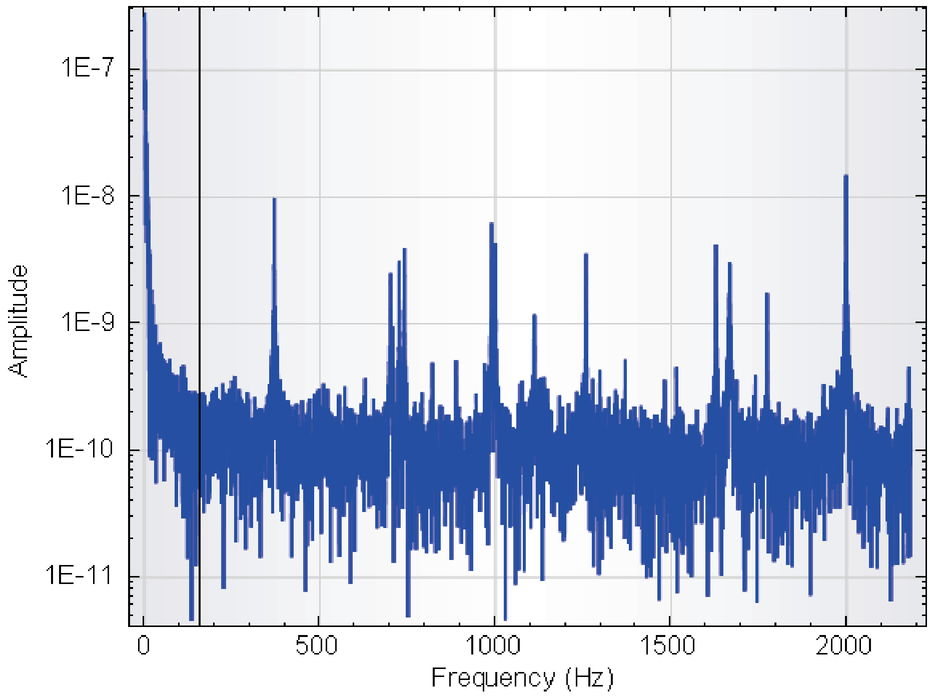


Figure 4.111 – Specifying the cutoff frequency for a low-pass or high-pass FFT filter

4.8.1.2.2 – Band pass and band stop filtering

For band pass or band stop filtering, two frequencies need to be specified in the FFT Smooth options panel (Frequency 1 and Frequency 2), as shown in Figure 4.112.

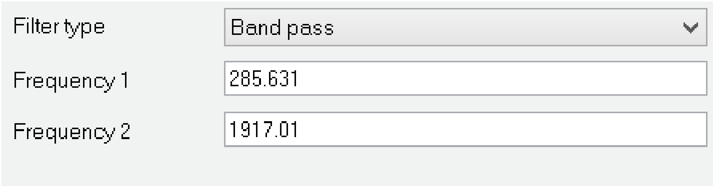


Figure 4.112 – Two frequencies need to be specified in the FFT Smooth options panel for band pass or band stop filtering

Note

Frequency 1 must be smaller than Frequency 2.

Alternatively, the mouse pointer can be used to select an area of the plot defining the frequency band for the band pass or band stop filter. Click the frequency domain plot at one of the required frequency limits and, while holding the mouse button, drag an area across the plot. Release the mouse button at the other frequency limit to define the frequency band to be used in the FFT Smooth filter (see Figure 4.113).

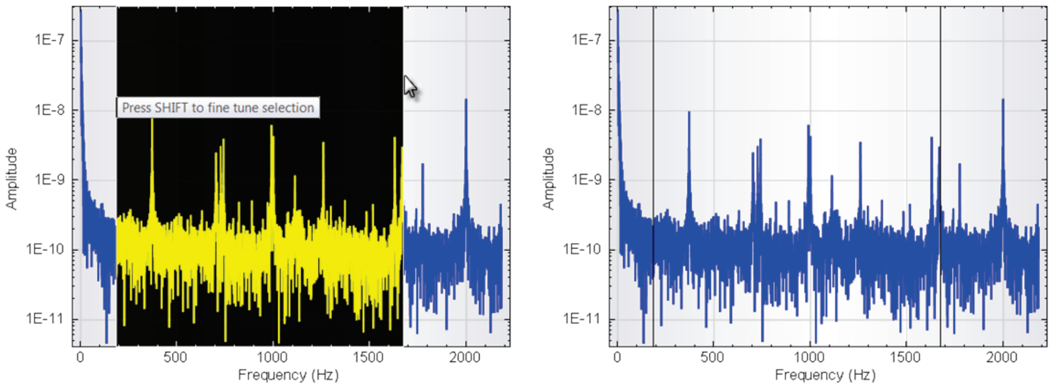


Figure 4.113 – The frequency band can be defined graphically by dragging an area across the plot

Two lines will be drawn on the plot to indicate the positions of the frequencies in the plot (see Figure 4.113). These values will also be shown in the FFT Smooth options.

Tip: use the Show coordinates option to help define the frequency or frequency range in the FFT Smooth analysis tool (see Figure 4.114).

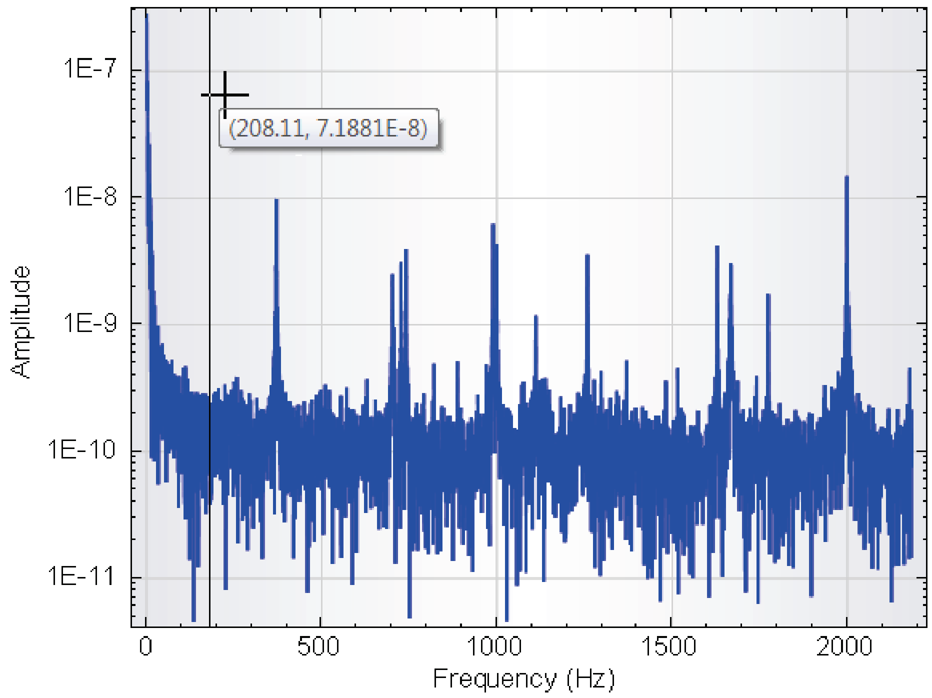


Figure 4.114 – Use the Show coordinates options to easily define the frequency or frequency range in the FFT Smooth analysis tool

The smoothed data will be automatically generated using the filtering conditions specified through the FFT Smooth options panel or graphically (see Figure 4.115).

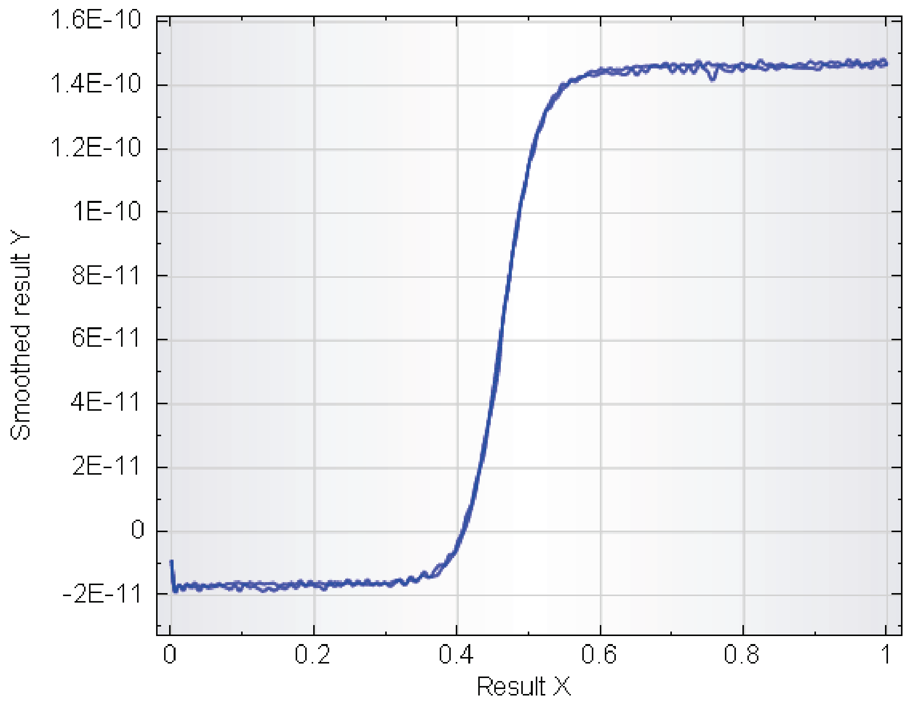


Figure 4.115 – The Smoothed data plot generated by the FFT Smooth tool

If required, a new frequency or frequency range can be selected by clicking the frequency domain plot again or by specifying a new frequency or frequency range in the FFT Smooth options panel. The data will be automatically re-filtered using the new settings.

4.8.1.3 – Baseline correction

The baseline correction can be used to fit a user-defined curve through measured data points to define a baseline and to automatically correct the data for it. Different forms of baselines can be defined in the software.

The baseline fit analysis tool can be added to any plot, using the right-click menu. This section provides details on how to use the Baseline correction tool.

Three different baseline corrections are possible:

- **Polynomial fixed order:** defines a polynomial baseline function of fixed order *n.* The number of markers required is *n+1.*
- **Polynomial maximum order:** defines a polynomial baseline function. The maximum polynomial order, *n,* is defined by the user. The polynomial order of the baseline curve is determined by the software, by minimization of the standard deviation values. The number of markers required is *n+1.*
- **Exponential:** defines an exponential baseline function. The equation used to determine the baseline curve can be modified using an optional offset value. The number of markers required is 4.

Double click on the demo file *Demo 10 - Differential pulse measurement* entry of the demo database to load it into the data explorer frame. This file contains data points recorded during a differential pulse voltammetry measurement (Zn^{2+}, Cd^{3+}, Pb^{2+} and Cu^{2+}, in acetate buffer).

Click the i vs E plot in the data explorer frame to display the data points from this demo file (see Figure 4.116).

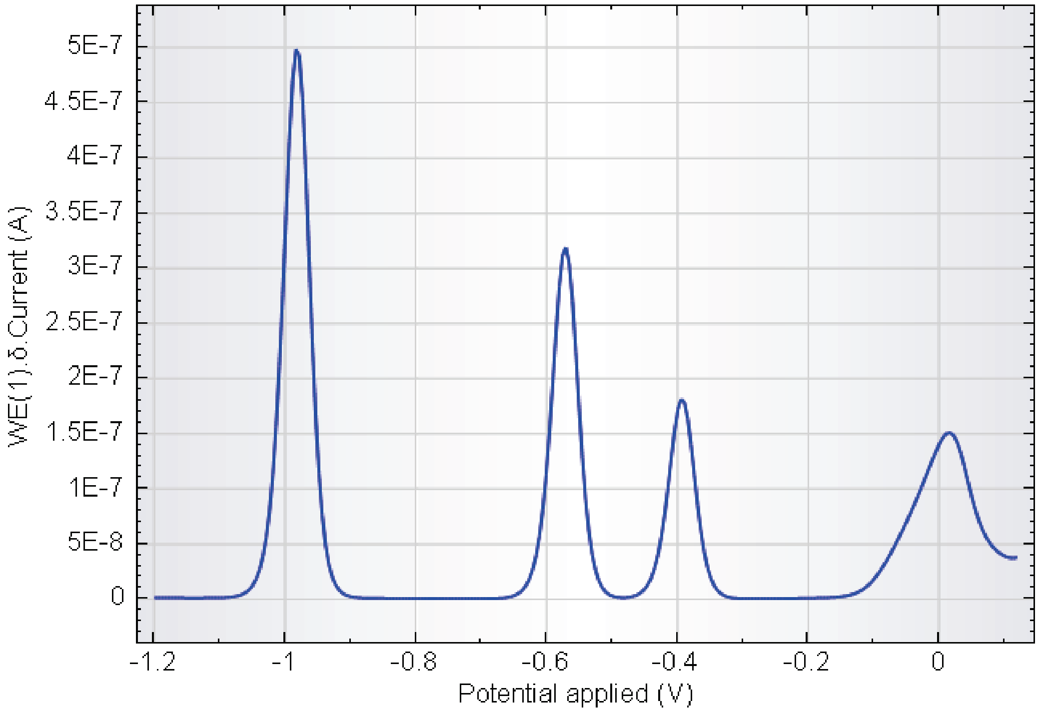


Figure 4.116 – The data points from Demo 10

Starting at a potential of -1.2 V, four peaks are visible in the data set. The last peak, located around 0 V does not have a linear baseline. The baseline fit tool can therefore be used to correct the data for this non-linearity.

To correct the data using a polynomial baseline fit, right-click the i vs E plot in the data explorer frame and, from the context menu, select the Add analysis – Baseline correction – Polynomial fixed order (see Figure 4.117).

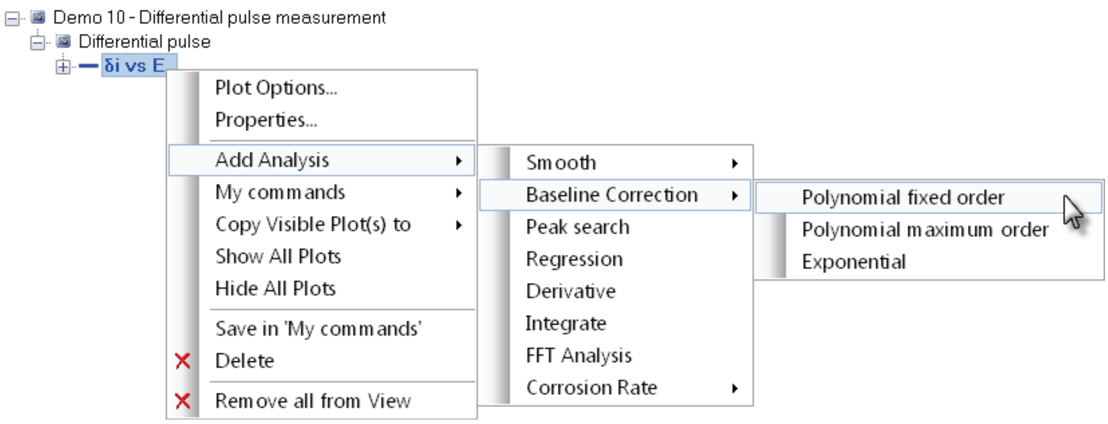


Figure 4.117 – Adding a baseline correction to the data

A new item, called Polynomial fixed order, will be added to the data set in the data explorer frame below the i vs E plot (see Figure 4.118).

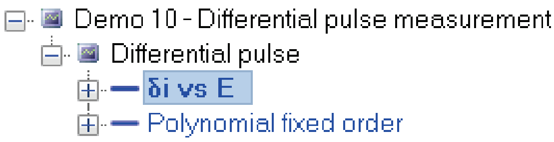


Figure 4.118 – The newly added Polynomial fit fixed order analysis item

Click the Polynomial fixed order analysis item. The mouse pointer will change to a cross and a new area will be displayed on the right-hand side of the plot (see Figure 4.119).

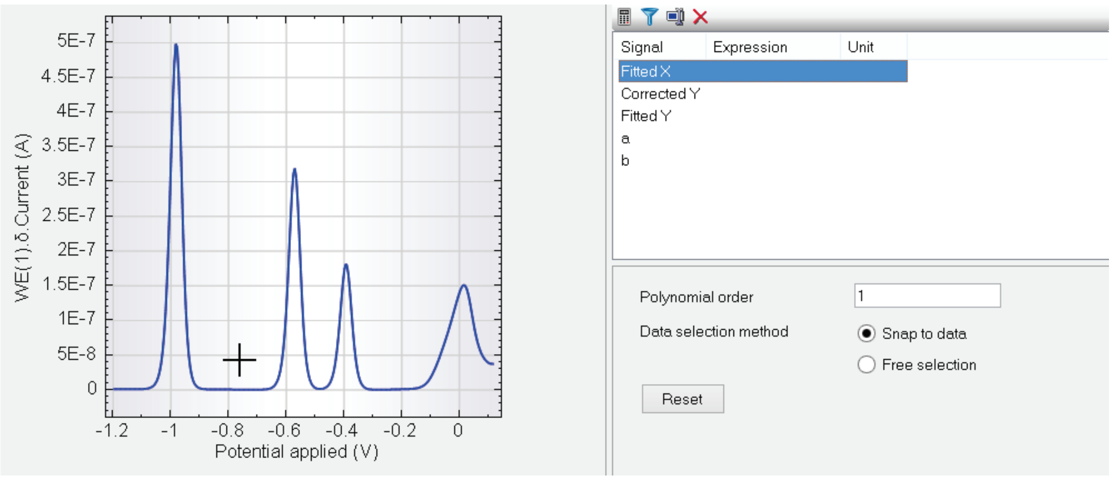


Figure 4.119 – Activating the baseline correction tool

The area located on the right of the 2D plot is used to set the parameters of the baseline fit. Set the Polynomial order to 4 for this example. The point selection style field should be set to Snap to data.

Note

When the Snap to data option is used, the software automatically positions the marker on the closets data point on the plot. Use the Free selection option to define points not on the curve.

Using the left mouse button, click a total of 5 measured data points in the 2D plot area. These points are used to define the points through which the baseline should pass. Since a baseline of 4th polynomial order is used, a minimum of 5 points are required, although more than 5 can be defined.

Figure 4.120 shows an example of marker location used to calculate the polynomial baseline. When the fifth marker is placed, the baseline will be calculated and plotted in the 2D plot area (see Figure 4.120).

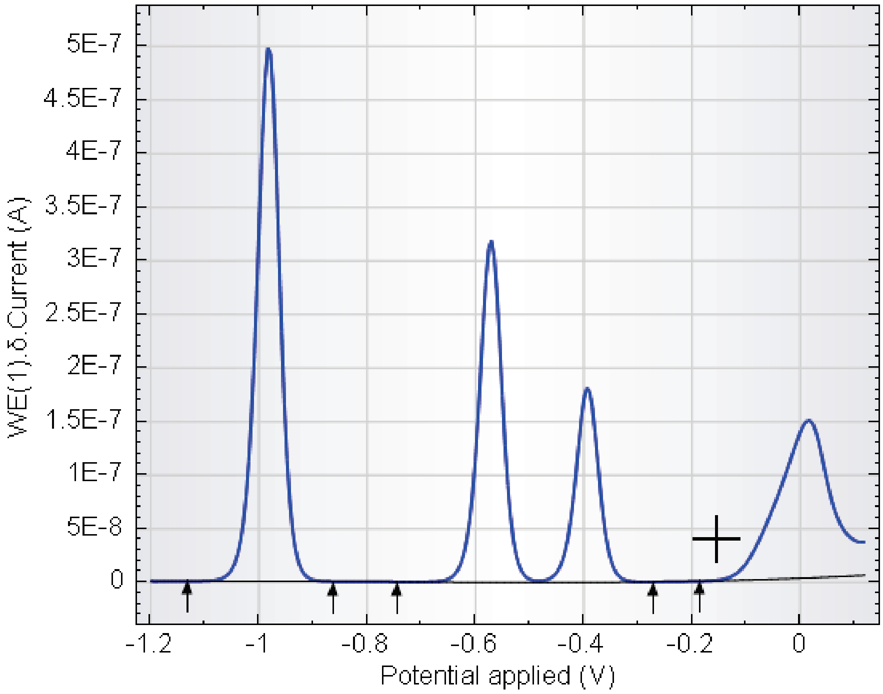


Figure 4.120 – Placing the markers to define the baseline

Note

The number of markers required to drawn a polynomial baseline is equal to the polynomial order +1.

The calculated baseline is not yet suitable for this experimental data and it requires additional fine tuning. This is possible in two different ways:

- By adding extra marker points
- By moving existing marker points

4.8.1.3.1 – Adding extra marker points

The first way to fine tune the baseline calculation is to add new markers to the plotted data. Figure 4.121 shows the same plot as in Figure 4.120, but with an extra marker located close to the positive end of the potential scan. When the new marker is placed, the baseline is recalculated and replotted in the 2D plot area.

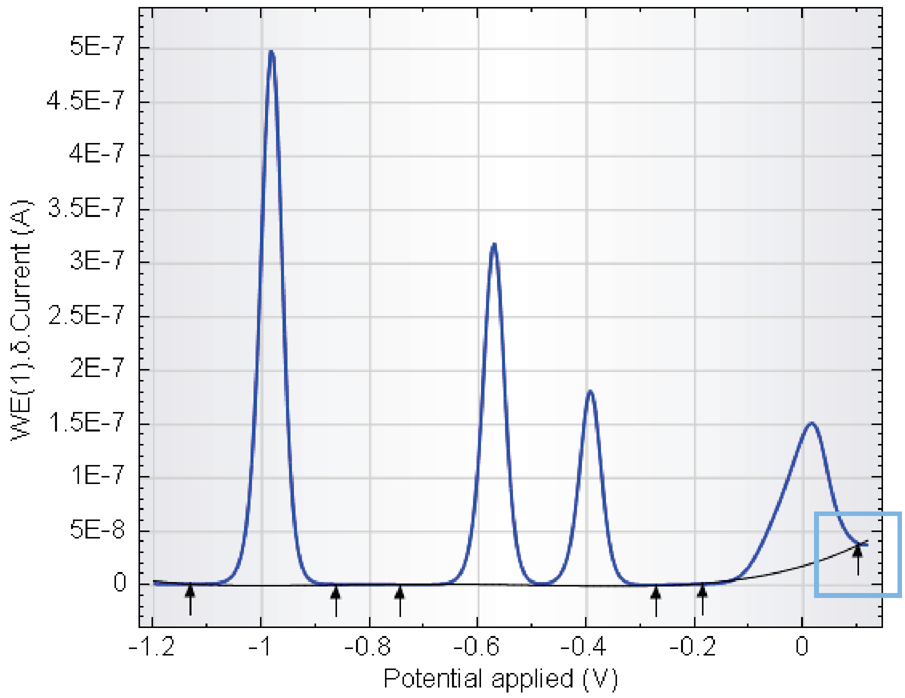


Figure 4.121 – Adding extra markers to the baseline allows to fine tune the calculation

The new marker allows for a better definition of the baseline at the positive end of the potential scan, compared to Figure 4.120. It is possible to keep adding markers to the baseline in order to improve the fit, if required.

Adding extra markers to a specific area of the curve increases the relative importance of that specific area of the plot in the baseline fit.

4.8.1.3.2 – Moving marker points

A second option that can be used to fine tune the calculated baseline is to move markers that are already placed on the plot. In order to do this, right-click on an existing marker point and select the Move option from the menu (see Figure 4.122).

Note

The same menu can be used to remove an existing marker point from the plot.

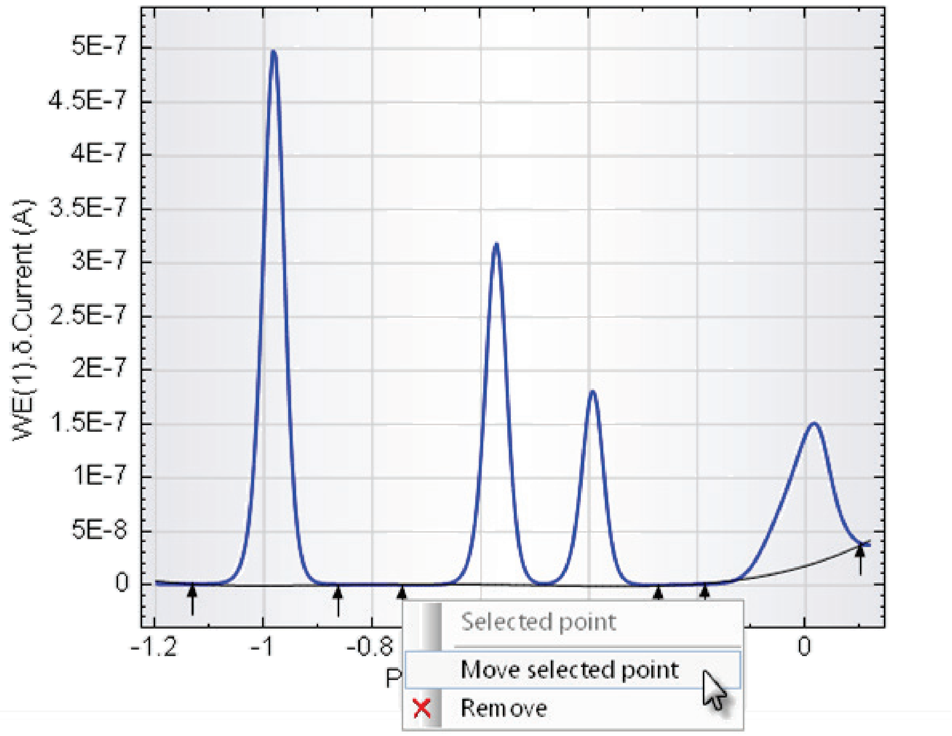


Figure 4.122 – Right-clicking an existing marker point allows you to move or remove the marker

After choosing the move option, a label showing the X and Y coordinates of the point will be displayed below the selected marker. Using the right and left key on the keyboard, it is possible to move the selected marker along the plot (see Figure 4.123).

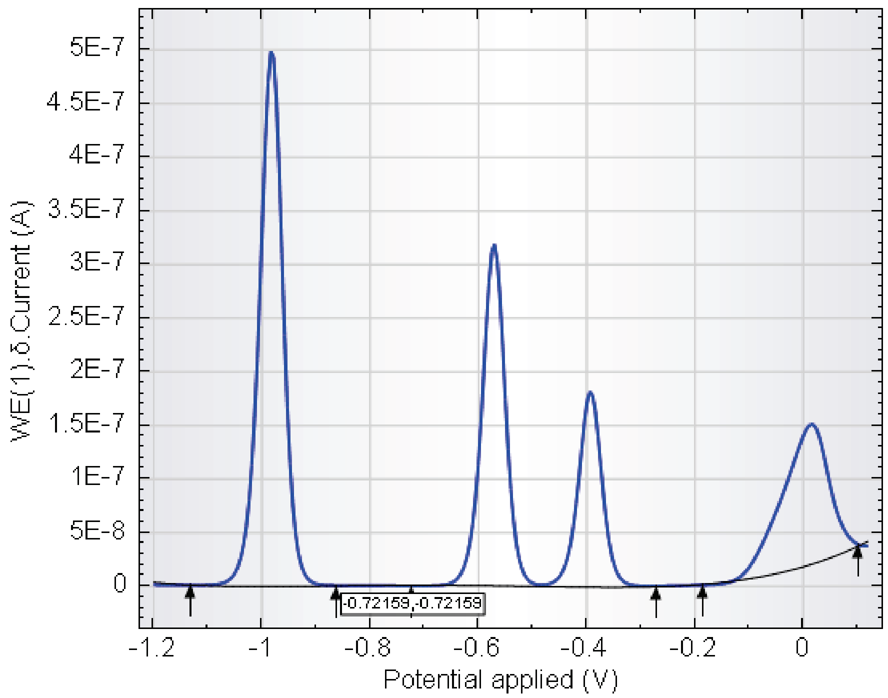


Figure 4.123 – Choosing the move option allows you to relocate the marker

When a suitable new position for the marker is located, press the Enter key on the keyboard to validate the new position. Click the plot to redefine the position of the marker for the baseline calculation. The baseline will be recalculated and replotted using the new set of markers (see Figure 4.124).

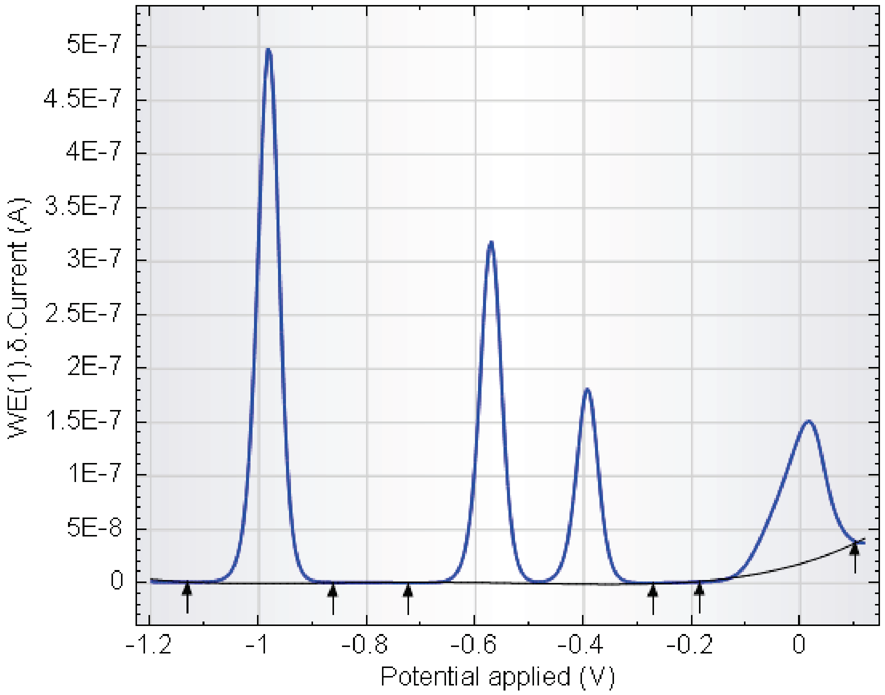


Figure 4.124 – The recalculated baseline

4.8.1.3.3 – Plotting the residual

When the baseline has been defined, it is possible to plot the original data corrected for the baseline. To do this, expand the Polynomial fixed order item in the data explorer frame and select the available Residual plot (see Figure 4.125).

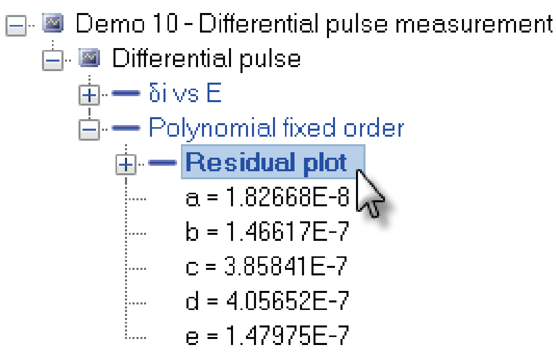


Figure 4.125 – Selecting the Residual plot generated by the baseline correction tool

Note

The coefficients listed below the Residual plot item, correspond to the coefficients used in the polynomial regression of the baseline. In this example, a fourth order polynomial fit was used, resulting in five coefficients ($y = a + bx + \cdots + ex^4$).

The data will be displayed in the 2D plot (see Figure 4.126).

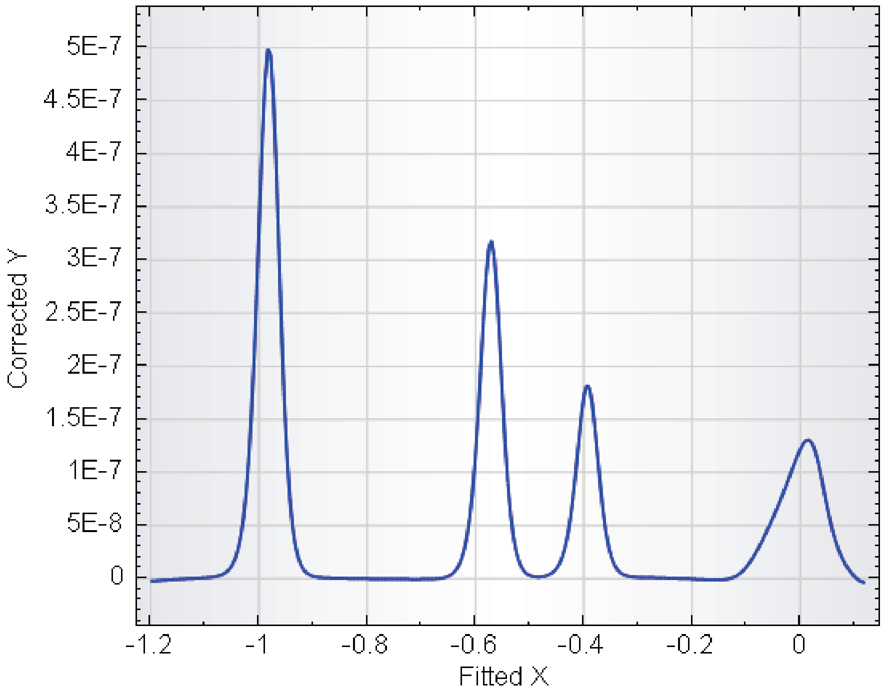


Figure 4.126 – The data point from Demo 10 after baseline correction

Note

The fitted Y values (corresponding to the baseline) and the corrected Y values (corresponding to the corrected data) are available in the data grid.

4.8.1.4 – Peak search

The peak search analysis tool can be used to locate and characterize peaks in an experimental curve. The peak search tool searches for peaks that fit the user-defined search criteria and displays information about the peaks in the Analysis view. NOVA offers two types of peak search modes: **Automatic** or **Manual**.

Double click on the demo file *Demo 14 - FC-(CH2)2-FC in ACN/CH2Cl2* entry of the demo database to load it into the data explorer frame. This file contains data points of a cyclic voltammetry staircase experiment recorded in aceto-nitrile/chloroform in the presence of 1,2-Diferrocenylethane, with TBu_4PF_6 as supporting electrolyte[32].

Click the i vs E plot in the data explorer frame to display the data points from this demo file (see Figure 4.127). Two anodic and two cathodic peaks are visible in the cyclic voltammogram.

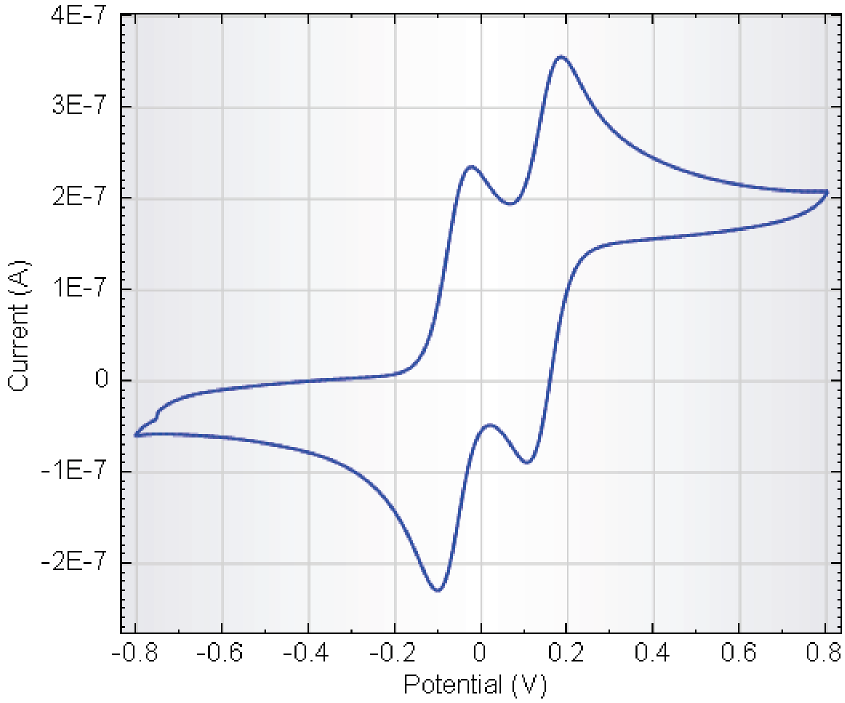


Figure 4.127 – The data from the Demo 14 data file

To use the peak search analysis tool, right-click the i vs E plot in the data explorer frame and, from the context menu, select the Peak search tool (see Figure 4.128).

[32] This data set is an imported GPES file.

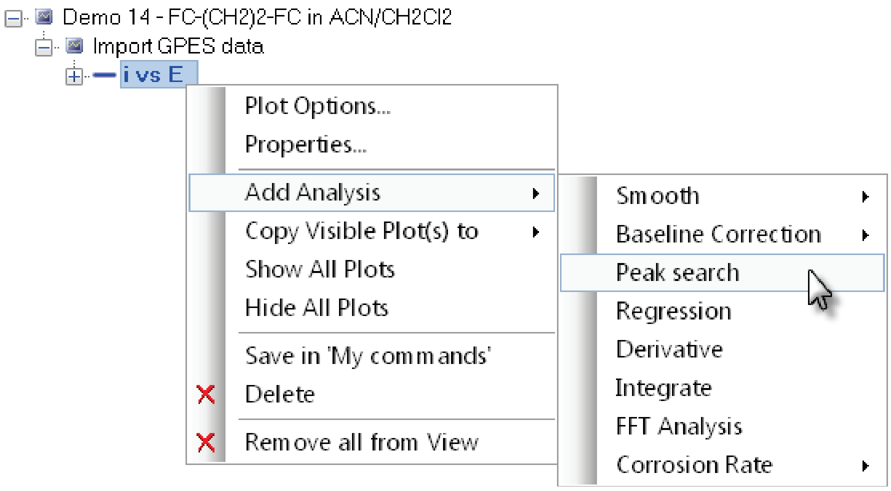


Figure 4.128 – Adding the peak search analysis tool to the data

A new item, called Peak search, will be added to the data set in the data explorer frame, below the i vs E plot (see Figure 4.129).

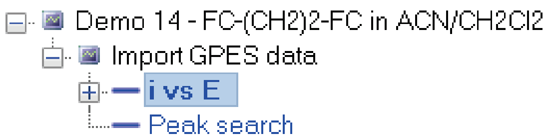


Figure 4.129 – The newly added Peak search analysis tool

Click the Peak search analysis item. A new area will be displayed on the right-hand side of the plot. This part of the view allows the definition of the selection criteria for the peak search tool (see Figure 4.130).

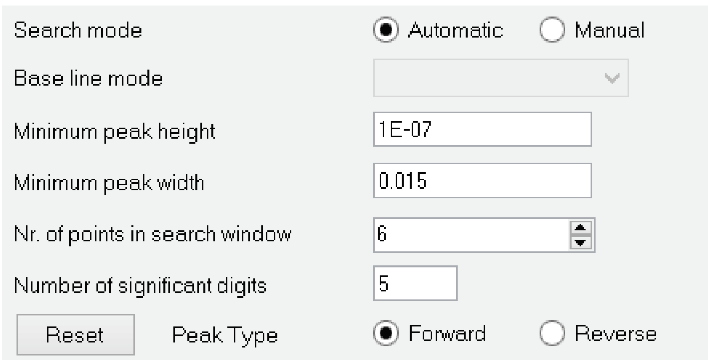


Figure 4.130 – The peak search selection criteria

The following selection criteria for the peak search tool are available:

- **Search mode**: automatic or manual.
- **Baseline mode:** defines the type of baseline used to determine the base of the peak. The **linear tangent baseline** is used when performing an automatic peak search. Other types of baselines are available in manual peak search.
- **Minimum peak height:** defines the minimum height of the peak, in Y-axis units (0 means that this selection criterion is not used).
- **Minimum peak width:** defines the minimum width of the peak, at half height, in X-axis units (0 means that this selection criterion is not used).
- **Nr. of points in search window:** this parameter defines the number of points that must be located above and below a zero crossing of the first derivative of the signal, in order to qualify as a peak. This setting is useful to discriminate between noise and real peaks[33].
- **Number of significant digits:** defines the number of significant digits used in the Analysis results frame.
- **Peak type:** forward or reverse. Using the forward setting, NOVA will search for *regular* peaks (anodic peak during the positive going scan or cathodic peak in the opposite direction). The reverse setting allows NOVA to search for peaks in the opposite direction.

4.8.1.4.1 – Automatic peak search

When the peak search analysis tool is added to the data as described in the previous section (see Figure 4.128), the calculation should be performed automatically, using the default settings. A total of four peaks should be detected, as shown in Figure 4.131.

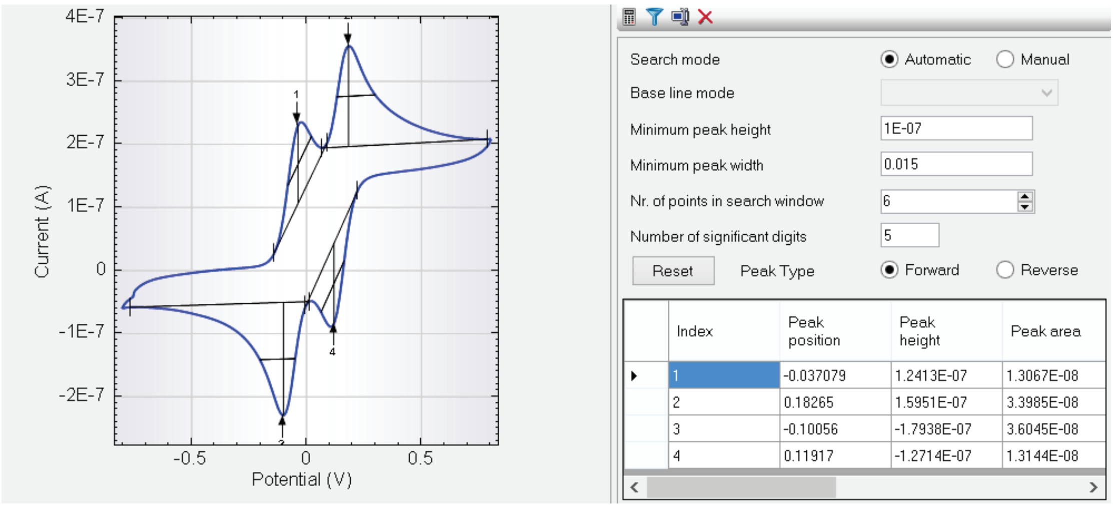


Figure 4.131 – The results of the automatic peak search

[33] This parameter is inactive in Manual search mode.

The table at the bottom right of the screen displays information for each peak. The peak position, peak height, peak area are listed, among other results. These results are also available in the data grid (see Figure 4.132).

Index	Peak position	Peak height	Peak area	Base start	Base end	Peak width half height	Peak (1/2)	Peak sum of derivatives
1	-0.037079	1.2413E-7	1.3067E-8	-0.13962	0.089874	0.10276	0.045033	3.5521E-6
2	0.18265	1.5951E-7	3.3985E-8	0.06546	0.78812	0.16615	0.050395	3.3802E-6
3	-0.10056	-1.7938E-7	3.6045E-8	-0.76462	0.011749	0.15401	-0.051147	3.85E-6
4	0.11917	-1.2714E-7	1.3144E-8	-0.0028992	0.22171	0.1009	-0.044652	3.7511E-6

Figure 4.132 – The results of the peak search analysis are displayed in the data grid

The following results are calculated by the peak search tool:

- **Index:** this is a unique label used to identify the peak in the curve.
- **Peak position:** X axis position of the maximum Y value with respect to the baseline, in X units.
- **Peak height:** maximum Y value with respect to the baseline, in Y units.
- **Peak area:** the geometric area located between the identified peak and the baseline, in units of X · Y.
- **Base start:** X axis position of the beginning of the baseline used to locate the peak, in X units.
- **Base end:** X axis position of the end of the baseline used to locate the peak, in X units.
- **Peak width half height:** the width of the peak, in X axis units at half the value of the peak height.
- **Peak (1/2):** the difference between peak position and the peak position at half height, in X axis units.
- **Peak sum of derivatives:** the sum of the absolute values of the maximum and the minimum in the derivative of the Y signal with respect to the X signal, in Y/X units.

Whenever one of the search criteria is updated in the software, the calculation is automatically updated, using the new, user-defined settings.

Changing the Minimum peak height to 1.5E-7 automatically updates the peak search results, as more peaks are found (see Figure 4.133).

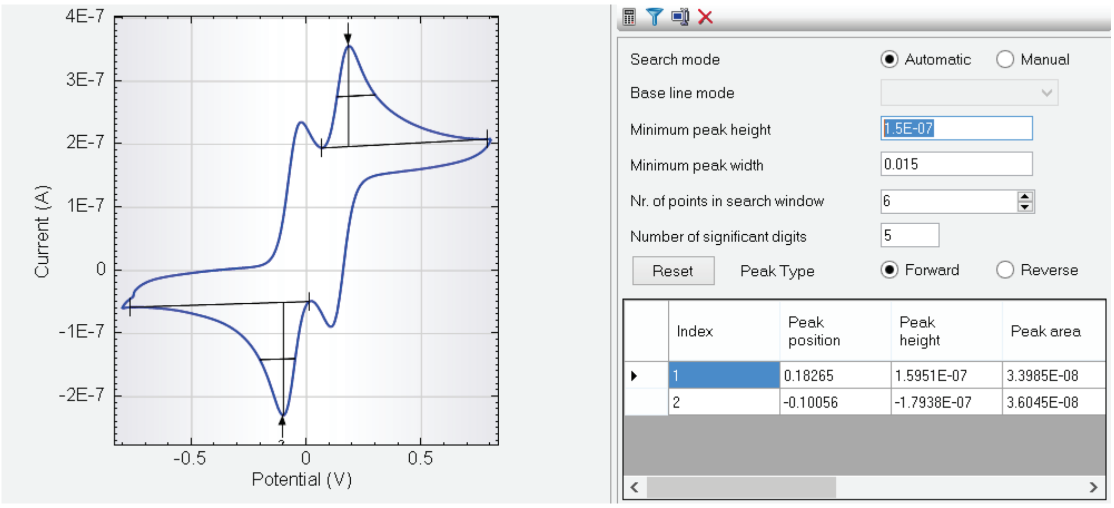


Figure 4.133 – Changing the peak search conditions automatically updates the results

4.8.1.4.2 – Manual peak search

Manual peak search allows for fine tuning of the search criteria and results. To perform manual peak search, switch to Manual search mode (see Figure 4.134).

Note

Switching search modes removes all the previously found peaks from the list.

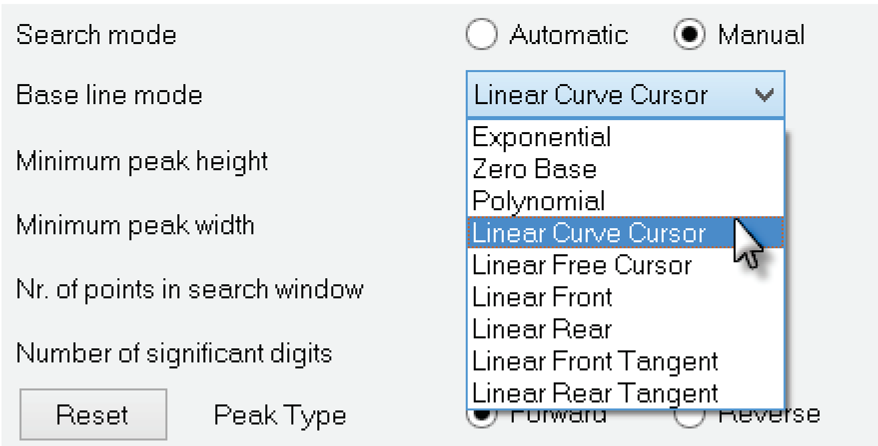


Figure 4.134 – In Manual search mode, the base line can be defined

In this case, the base line mode selection will become available (see Figure 4.134).

Note

The peak area is calculated only when the Linear Curve Cursor or the Polynomial methods are used.

To perform manual peak search, set the required search criteria and choose the base line mode from the drop-down list. A total of nine different baseline types are available in NOVA (see Figure 4.134):

- **Exponential:** this option uses an exponential baseline in the determination of the peaks. To define the baseline, click on the plot area. The software automatically connects the initial point to the nearest data point. *While holding the mouse button pressed*, drag the mouse across the plot area to define the baseline. When the mouse button is released, the final X position of the mouse pointer is used to define the X position of the final point of the baseline, on the curve (see Figure 4.135).

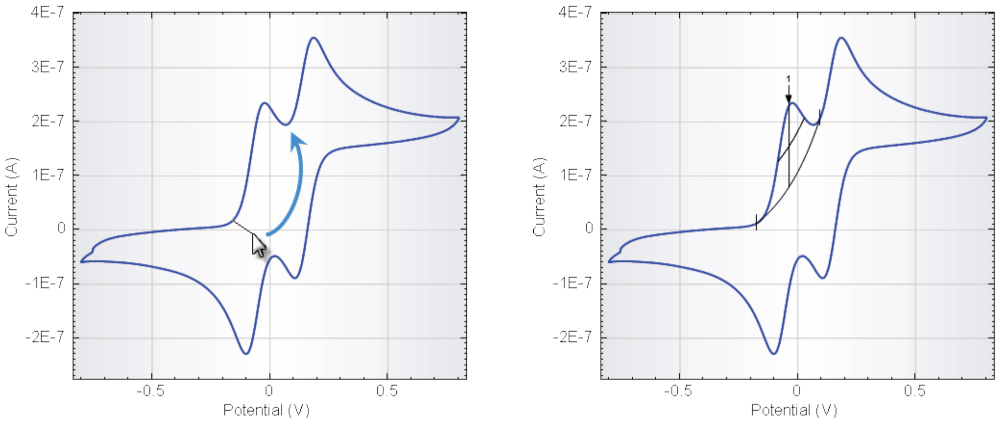


Figure 4.135 – Defining an exponential baseline for the peak (left – location of the first point, right – location of the second point and drawing of the baseline)

- **Zero Base:** no baseline is used in the determination of the peak. Click anywhere on the plot and *while holding the mouse button*, drag the mouse across the plot area to define the search window on the X axis. Using this baseline mode, the data point on Y axis, with the highest absolute value, located within the range defined on the X axis is used as a peak (see Figure 4.136).

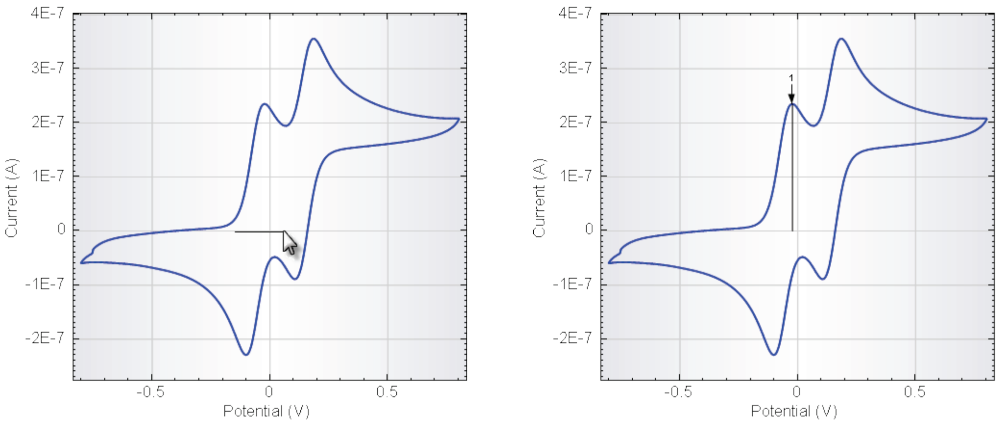


Figure 4.136 – Using the zero base method to specify the base line

Note

The zero base search method locates the absolute maximum value of the curve in the curve segment closest to the **first point** defining the search window.

- **Polynomial:** this baseline uses a polynomial function in the determination of the peaks. The polynomial baseline is constructed by clicking two or more points on the plot. These waypoints do not have to be on the curve itself. When the last point has been defined, press the **Enter key** on the keyboard to perform the analysis with the defined baseline (see Figure 4.137).

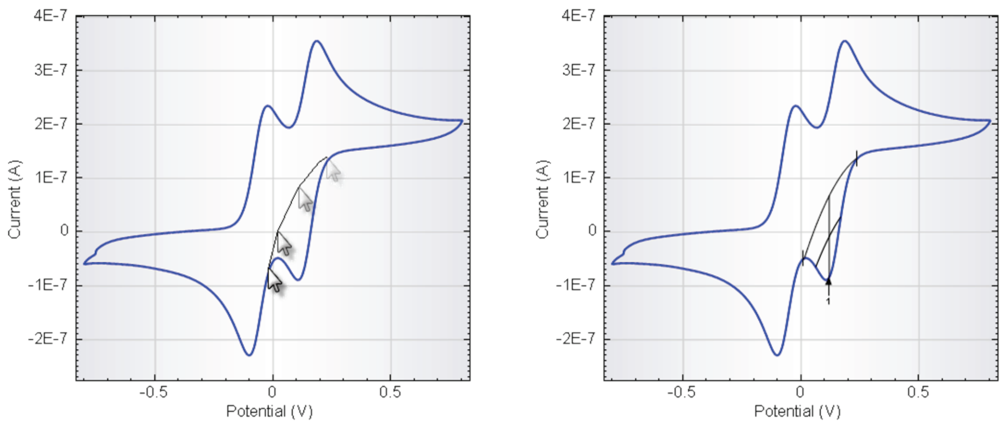


Figure 4.137 – Using the polynomial baseline mode (left – specification of the waypoints for the polynomial baseline, right – pressing Enter validates the polynomial baseline)

Note

The polynomial search method locates the peaks in the curve segment closest to the **first point** defining the polynomial baseline.

- **Linear Curve Cursor:** this option uses a linear baseline in the determination of the peaks. To define the baseline, click on the plot area. The software automatically connects the initial point to the nearest data point. *While holding the mouse button pressed*, drag the mouse across the plot area to define the baseline. When the mouse button is released, the final X position of the mouse pointer is used to define the X position of the final point of the baseline, on the curve (see Figure 4.138).

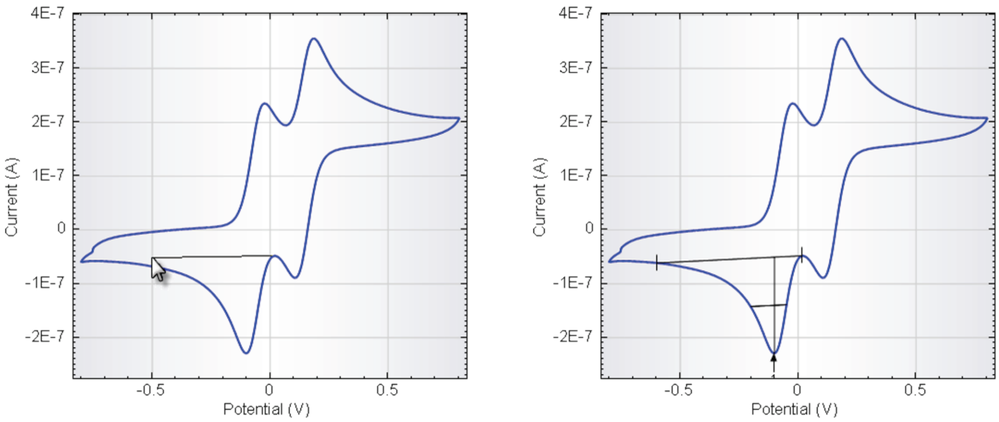


Figure 4.138 – Using the Linear curve cursor method to specify the baseline (left – location of the first point, right – location of the second point and drawing of the baseline)

- **Linear Free Cursor:** this option uses a linear baseline in the determination of the peaks. To define the baseline, click on the plot area and *while holding the mouse button pressed*, drag the mouse across the plot area. When the mouse button is released, the final X,Y position of the mouse pointer is used to define the final point of the baseline (see Figure 4.139).

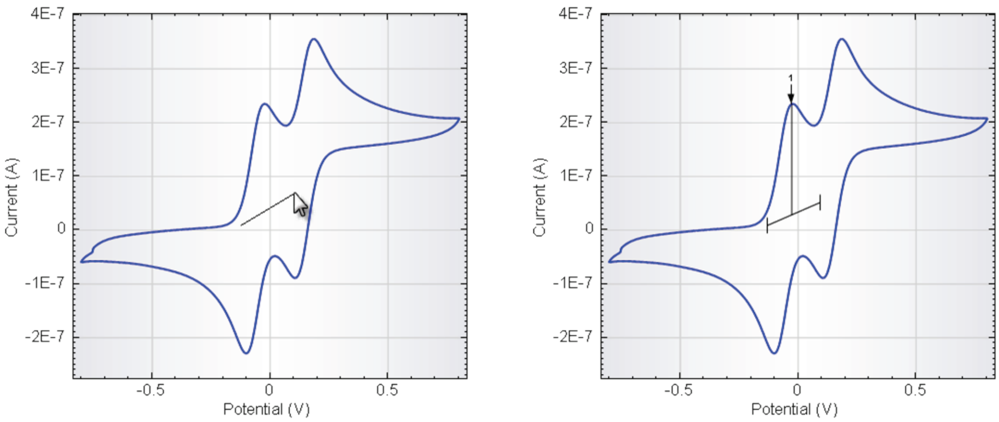


Figure 4.139 - Using the Linear free cursor method to specify the baseline (left – location of the first point, right – location of the second point and drawing of the baseline)

- **Linear Front:** this option finds peaks by extending a tangent baseline located in front of the peak. To define the baseline, click on the plot area. The software automatically connects the initial point to the nearest data point. *While holding the mouse button pressed*, drag the mouse across the plot area to define the baseline. When the mouse button is released, the final X position of the mouse pointer is used to define the X position of the final point of the baseline, on the curve. The tangent is then extended frontwards and the peak is located (see Figure 4.140).

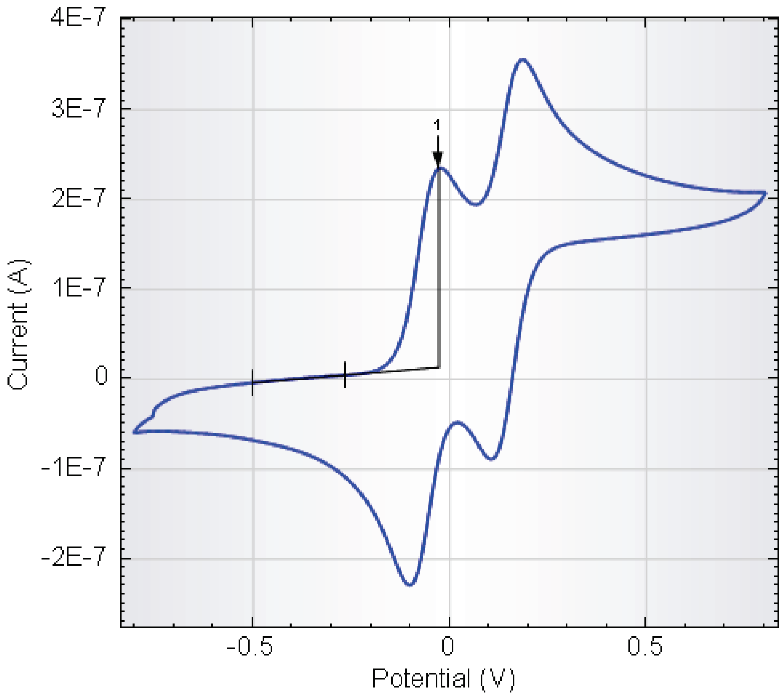


Figure 4.140 – Using the Linear front baseline search method

Note

The base points used to specify the tangent baseline are indicated by the small vertical lines on the plot (see Figure 4.140).

- **Linear Rear:** this option finds peaks by extending the baseline located after the peak. To define the baseline, click on the plot area. The software automatically connects the initial point to the nearest data point. *While holding the mouse button pressed*, drag the mouse across the plot area to define the baseline. When the mouse button is released, the final X position of the mouse pointer is used to define the X position of the final point of the baseline, on the curve. The tangent is then extended backwards and the peak is located (see Figure 4.141).

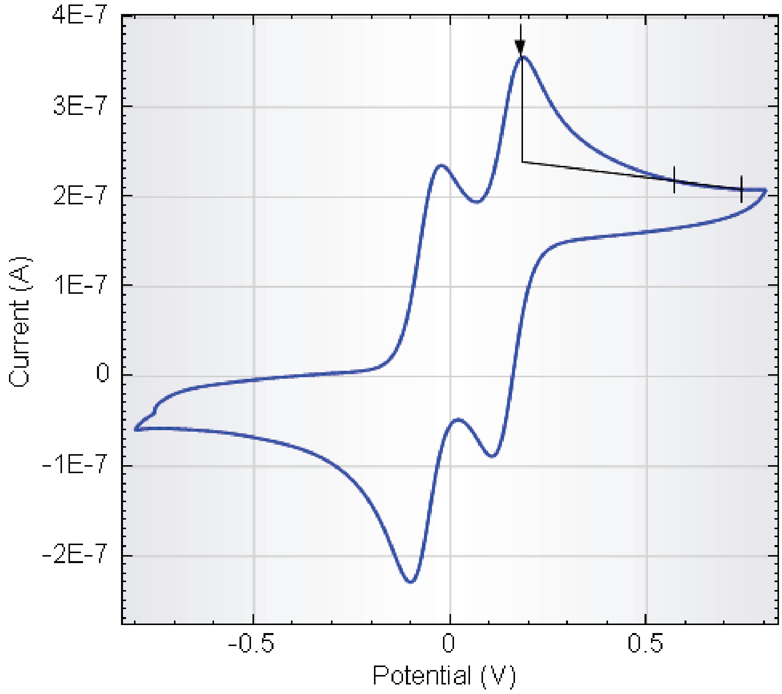


Figure 4.141 - Using the Linear rear baseline search method

Note

The base points used to specify the tangent baseline are indicated by the small vertical lines on the plot (see Figure 4.141).

- **Linear Front Tangent:** this option finds peaks by extending the baseline located in front of the peak. The baseline is defined by clicking on the plot area and, *while holding the mouse button pressed*, dragging the mouse across the plot area, drawing a straight line. The software automatically connects the baseline to the curve at the data point for which the first derivative is the closest to the slope of drawn baseline (see Figure 4.142).

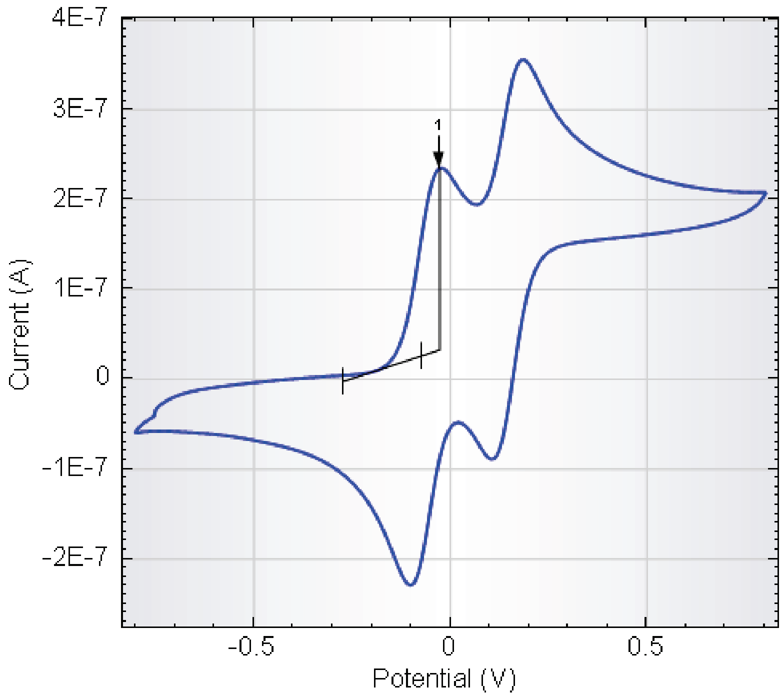


Figure 4.142 – Using the Linear front tangent baseline search method

Note

The base points used to specify the tangent baseline are indicated by the small vertical lines on the plot (see Figure 4.142).

- **Linear Rear Tangent:** this option finds peaks by extending the baseline located after the peak. The baseline is defined by clicking on the plot area and, *while holding the mouse button pressed*, dragging the mouse across the plot area, drawing a straight line. The software automatically connects the baseline to the curve at the data point for which the first derivative is the closest to the slope of drawn baseline (see Figure 4.143).

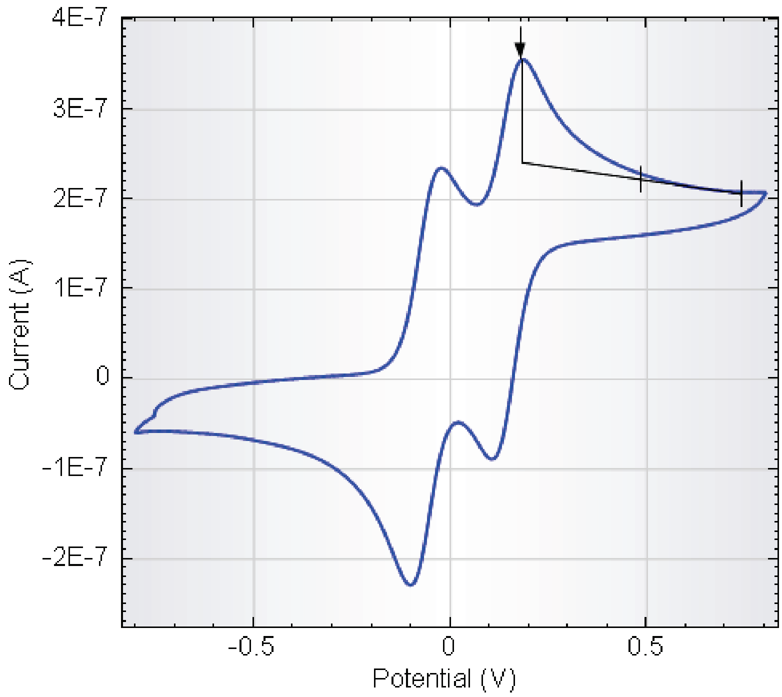


Figure 4.143 - Using the Linear rear tangent baseline search method

Note

The base points used to specify the tangent baseline are indicated by the small vertical lines on the plot (see Figure 4.143).

Use the manual peak search function in combination with the Show coordinated option (available from the right-click menu – see section 4.7.3). This will display the X and Y coordinates of the mouse pointer, while the baseline is defined, making it easier to fine tune the position of the peak (see Figure 4.144).

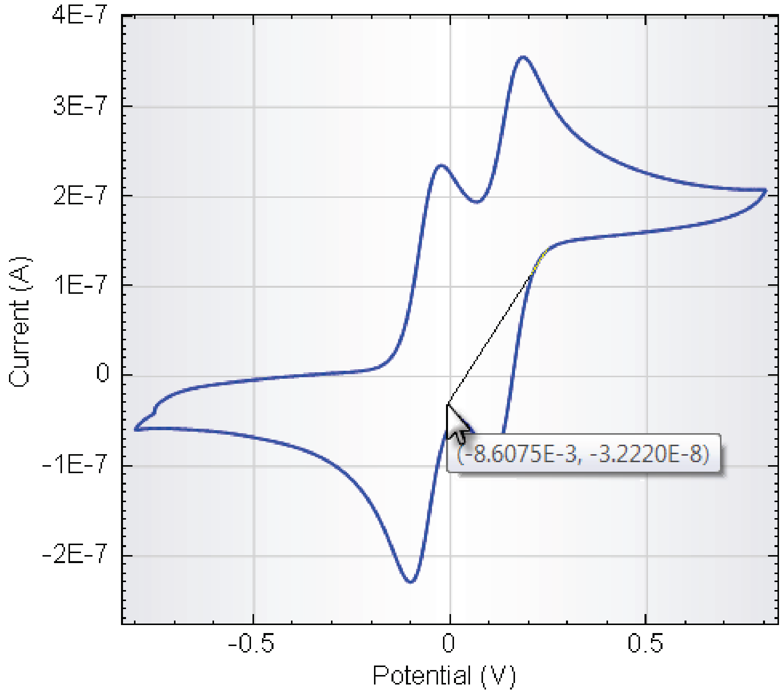


Figure 4.144 – Fine tuning the markers of the base line using the Show coordinates option in combination with the manual peak search tool

It is important to be able to fine tune the base line used in the manual peak search, after the search has been performed. It is possible to redefine the position of the left and right markers defining the base line. To do this, right-click the peak label and select either the Move left base point or the Move right base point, to redefine the left and right markers, respectively (see Figure 4.145).

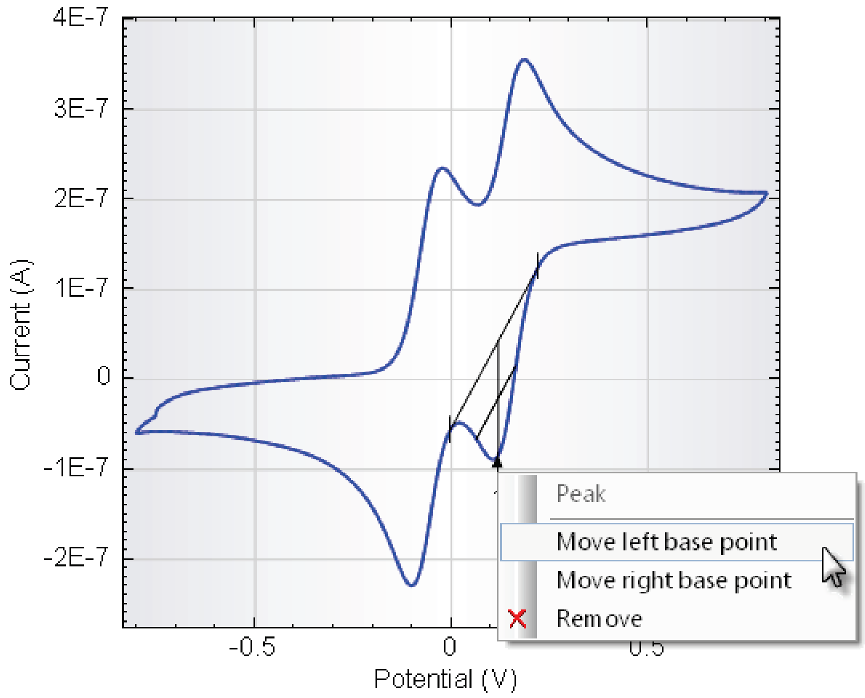


Figure 4.145 – Choosing the Move left base point option allows you to move the base line marker

A label will display the coordinates of the base point on the plot (Figure 4.146).

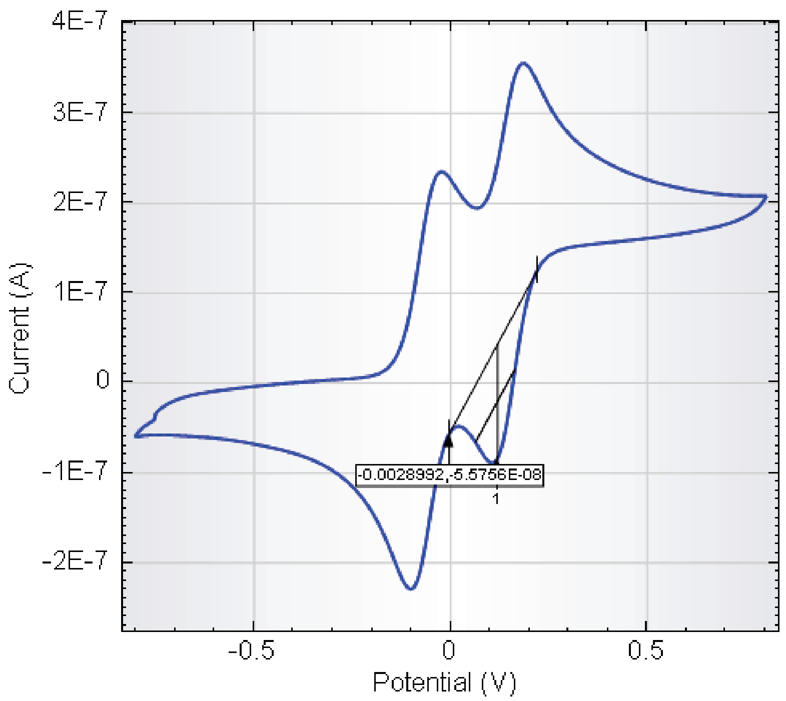


Figure 4.146 – The selected base point can be moved using the left and right key

Using the left and right arrow key on the keyboard, it is possible to move the marker point to the left and to the right. The base line used in the peak determination will be updated in real time while the marker is moved. When a convenient position has been found, **press the Enter key** on the keyboard to redefine the peak. The results of the peak search calculation will be updated, using the new base line.

Note

To clear the Analysis results frame of previous data, click the Reset button located in the analysis frame (see Figure 4.147).

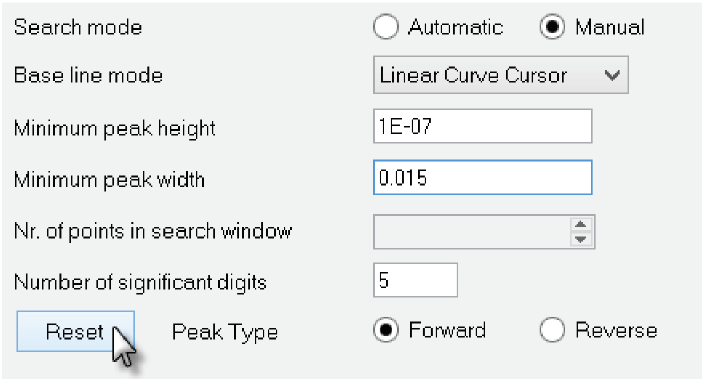


Figure 4.147 – Pressing the Reset button removes previous results from the analysis results frame

Note

The results of the Peak search are stored in the data grid.

4.8.1.5 – Regression

The regression analysis tool can be used to perform different regression calculations on measured data points. A wide range of functions can be used in this type of calculation:

- **Linear no offset:** performs a linear regression using the $y = ax$ equation.
- **Linear:** performs a linear regression using the $y = ax + b$ equation.
- **Polynomial fixed order:** this calculation uses a n^{th} order polynomial function in the regression calculation. The value of n is defined by the user.
- **Polynomial maximum order:** this calculation uses all the polynomial functions up to a maximum of n, defined by the user. The regression providing the smallest χ^2 (Chi-squared) is automatically selected by the software.

- **Exponential no offset:** this regression calculation is performed using the equation $y = be^{cx}$.
- **Exponential:** this regression calculation is performed using the equation $y = a = be^{cx}$.
- **Circle:** this regression calculation is performed using the equation of a circle.

The regression tool can be added to a measurement using the right-click menu and by selecting the Add analysis – Regression item from the context menu (see Figure 4.148).

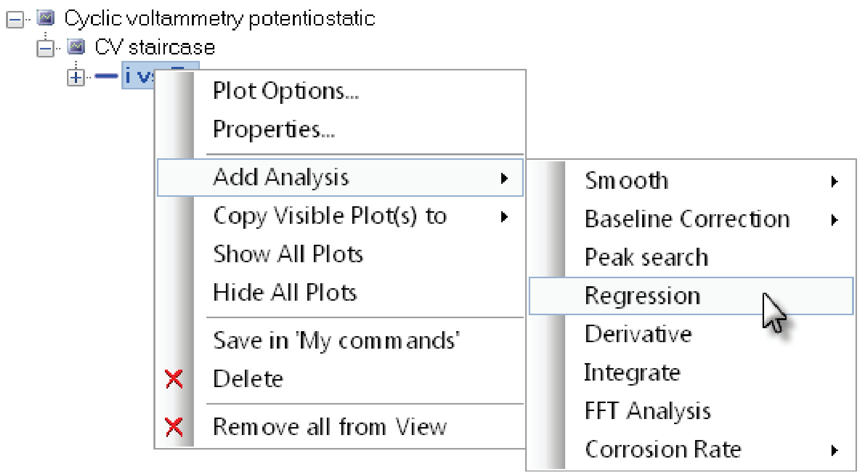


Figure 4.148 – The regression tool can be added to a plot using the right-click menu

Double click on the demo file *Demo 04 - Hydrodynamic LSV with increasing rotation rate* entry of the demo database to load it into the data explorer frame. This file contains data points recorded at different rotation rates with the Autolab RDE in a 0.05 M $[Fe(CN)_6]^{4-/3-}$ in 0.2 M NaOH solution.

The data file contains a total of six linear sweep voltammograms (see Figure 4.149).

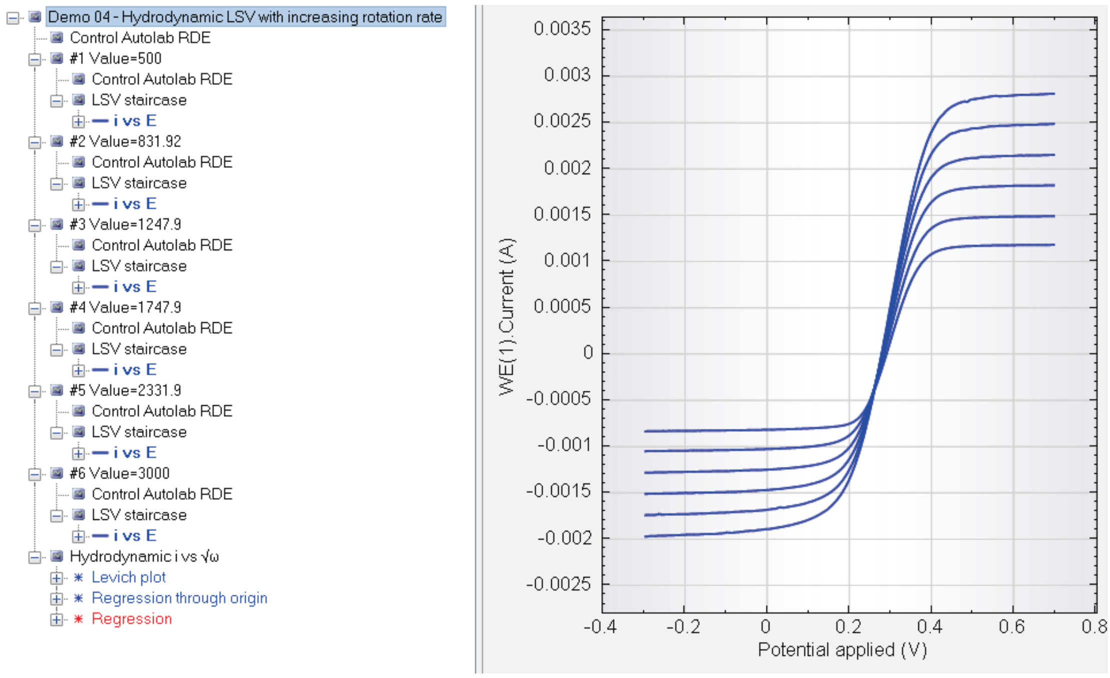


Figure 4.149 – The data from the Demo 04 data file

This demo file also includes an additional item, located at the end of the file in the data explorer frame, called *Hydrodynamic i vs* $\sqrt{\omega}$. This analysis item is designed to perform a simple Levich analysis on data recorded using forced-convection. This item automatically generates a Levich plot and performs two linear regressions on the plotted data, using the *linear* and the *linear no offset* regression methods (see Figure 4.150).

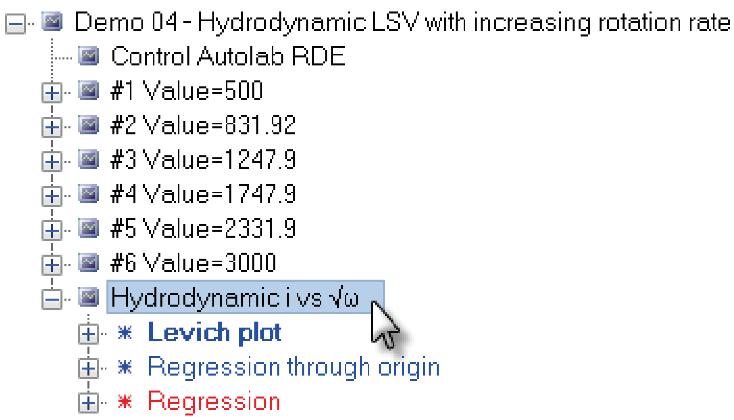


Figure 4.150 – The *Hydrodynamic i vs* $\sqrt{\omega}$ analysis tool automatically adds two regression lines to the Levich plot

Click the *Regression* item in the data explorer. The Levich plot will be displayed in the plot area, along with the linear regression line (see Figure 4.151).

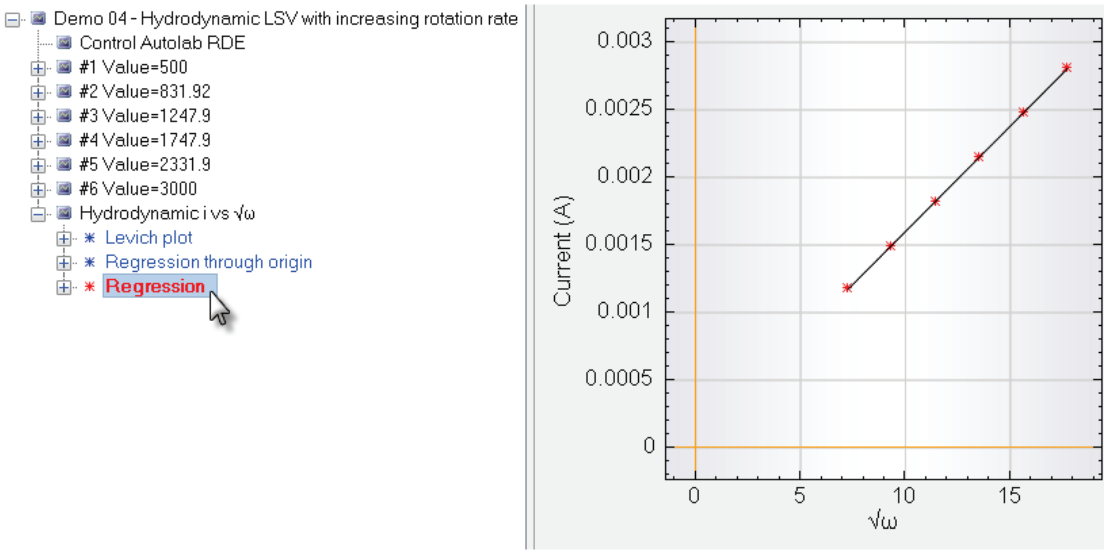


Figure 4.151 – Clicking the Regression item in the explorer view displays the Levich plot and the regression line

Clicking this regression item also displays the parameters of the regression analysis tool on the right-hand side of the plot (see Figure 4.152).

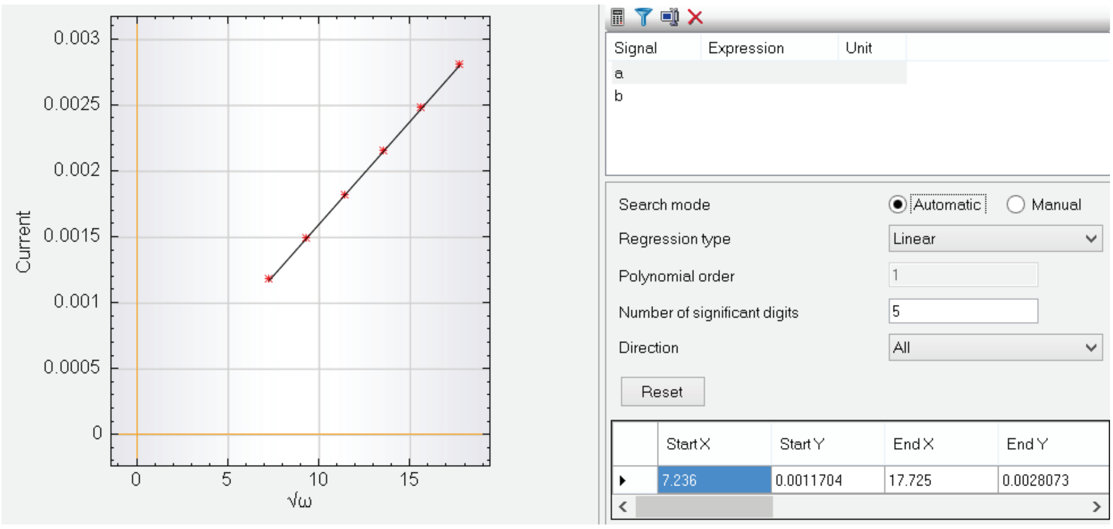


Figure 4.152 – The parameters of the Regression analysis tool are located on the right-hand side of the plot

The regression tool has a number of parameters that can be defined in the control interface:

- **Search mode:** Automatic or Manual. When the Automatic mode is used, the whole data set is used in the regression. Using the manual search mode lets the user define a window in which to use the regression calculation. The window is determined by clicking and holding the left mouse button while moving the mouse left or right.
- **Regression type:** Defines the type of regression.

- **Polynomial order:** Defines the order of a polynomial regression (this parameter is only available when using the polynomial regression).
- **Number of significant digits:** Defines the number of significant digits used in the Analysis results frame.
- **Direction:** from All, Forward and Reverse. **All** will apply the regression calculation to the whole data set (in the case of a cyclic voltammogram, both the positive going sweep and the negative going sweep will be fitted). **Forward** will only apply the calculation to the points from the positive going scan while **Reverse** will apply the fit only to the points from the negative going scan.

The linear regression confirms the linear relationship between the square root of the angular frequency, $\sqrt{\omega}$ and the anodic limiting current. The details of the linear regression are displayed in the data explorer frame (see Figure 4.153).

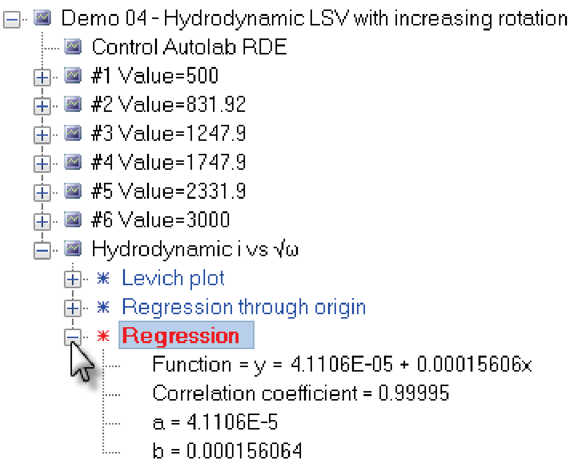


Figure 4.153 – The results of the linear regression are displayed in the data explorer

The automatic regression tool does not extend the calculated curve beyond the experimental data points. For plotting purposes, it might however be useful to extend the regression line through the origin of the plot. To do this, switch to Manual search mode (see Figure 4.154).

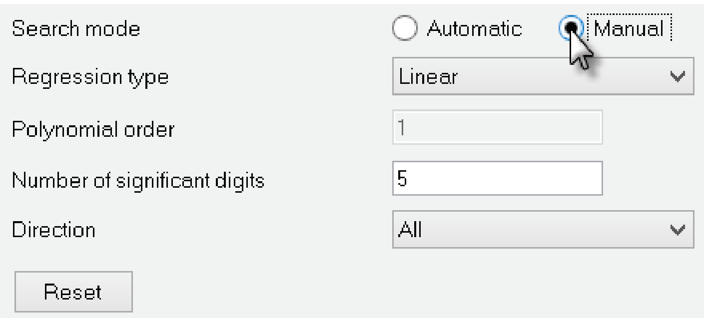


Figure 4.154 – Using the manual regression search mode

Note

Switching from Automatic to Manual search mode removes the results of the previous linear regression.

While holding the left mouse button, drag the mouse pointer across the 2D plot to define the region to use for the regression calculation (see Figure 4.155).

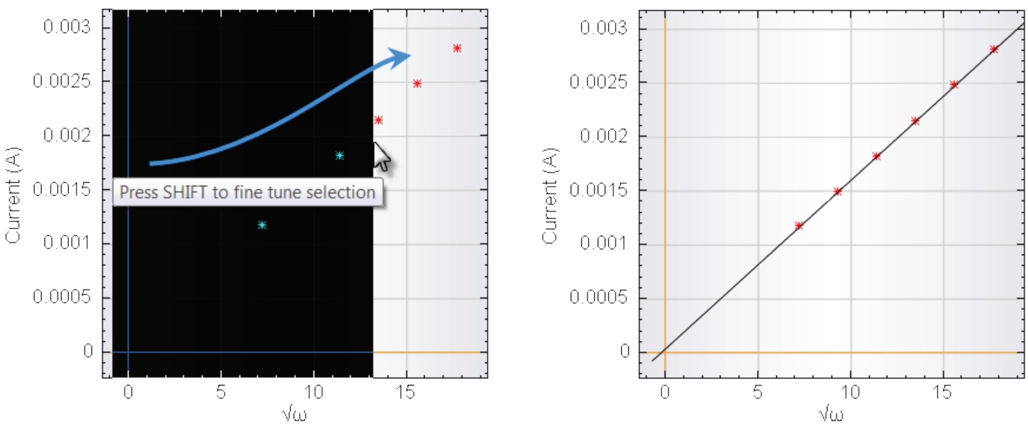


Figure 4.155 – Defining the region to use in the manual regression

Once the search region has been defined, the calculation is performed automatically using the settings defined in the control interface. Using this approach, the regression line can be extended through the origin of the plot as shown in Figure 4.155.

If the shift key is pressed while the search window is defined, it is possible to fine tune the search window (see Figure 4.156).

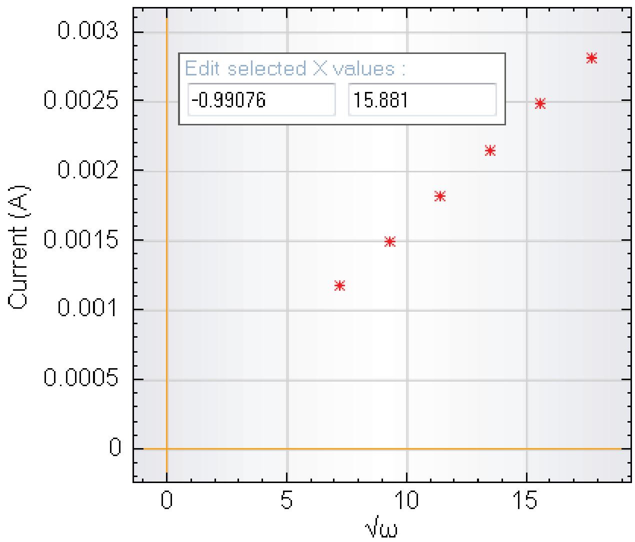


Figure 4.156 – The shift key allows to fine tune the search window

Note

The results of the Regression are stored in the data grid.

4.8.1.6 – Derivative

The derivative analysis tools can be used to calculate the first derivative of a plot. This tool can be added to any Y vs X plot in the data analysis view.

Double click on the demo file *Demo 19 – Cyclic voltammetry Fe^{2+}/Fe^{3+}* entry of the demo database to load it into the data explorer frame. This file contains data points of a cyclic voltammetry staircase experiment recorded in a 0.05 M $[Fe(CN)_6]^{4-/3-}$ in 0.2 M NaOH solution. The number of scans is three.

Click the i vs E plot in the data explorer frame to display the data points from this demo file (see Figure 4.157). A typical reversible electron transfer behavior is recognizable in the cyclic voltammogram.

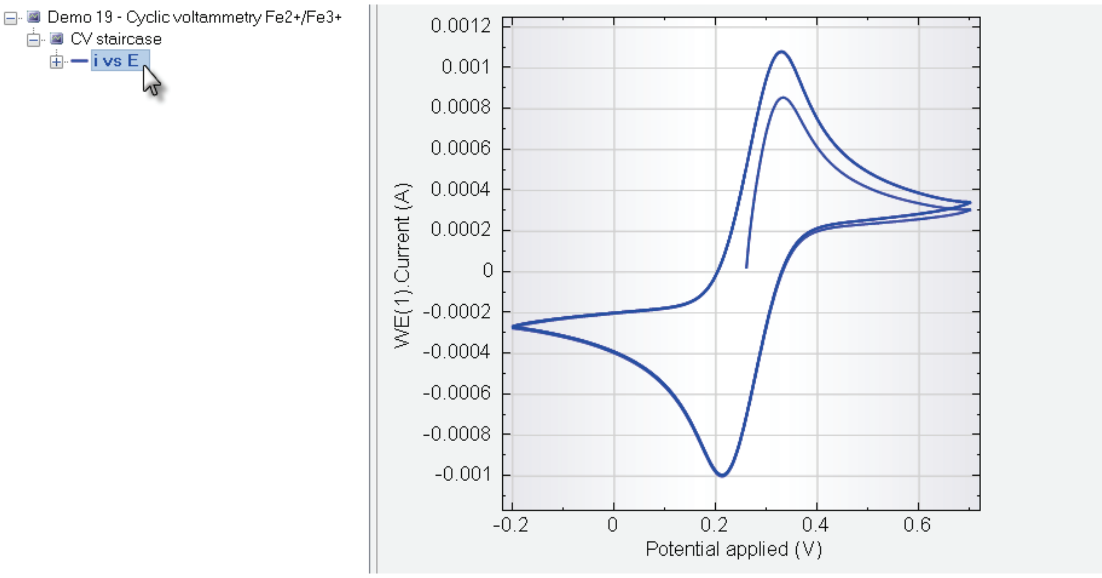


Figure 4.157 – The Demo 19 data file

Right click the CV staircase item in the data explorer frame, and select the i vs t plot from the plot context menu (see Figure 4.158).

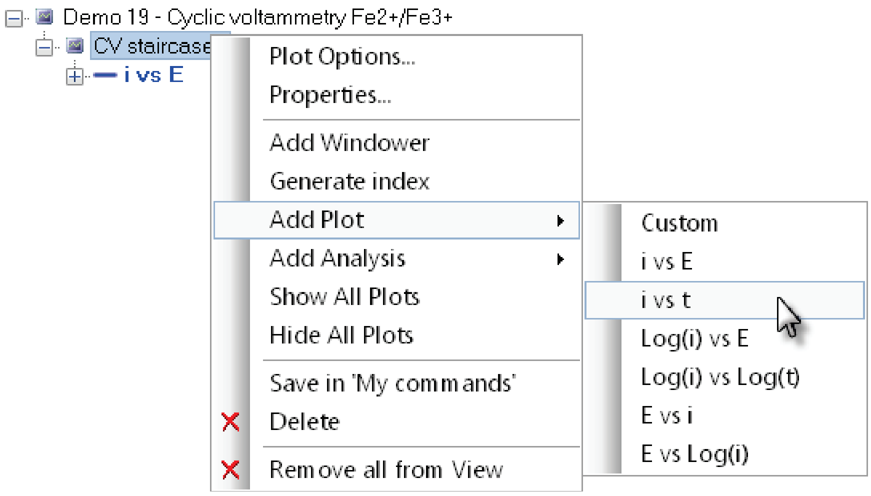


Figure 4.158 – Adding the i vs t plot to the data set

A new plot, showing the current vs time recorded during this experiment will be added to the data set. Click this new plot to display the plot in the analysis view (see Figure 4.159).

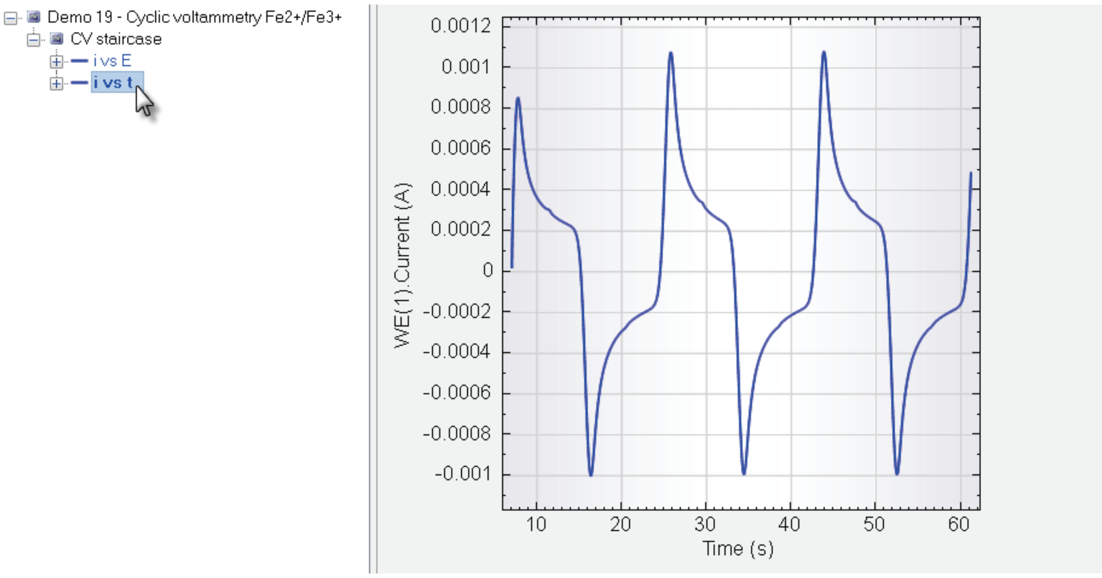


Figure 4.159 – The i vs t plot added to the data set

To calculate the derivative of current vs time, right-click the i vs t item in the data explorer and select the Derivative tool from the Add Analysis menu (see Figure 4.160).

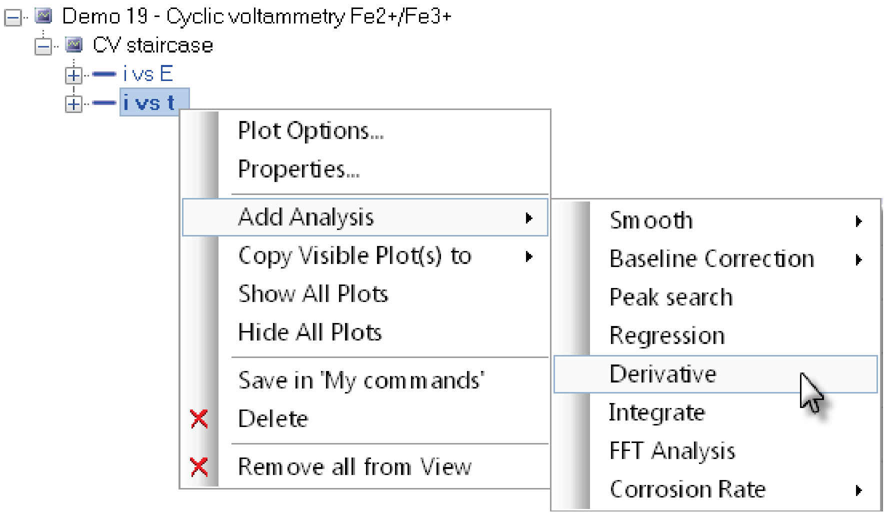


Figure 4.160 – Adding the Derivative tool to the i vs t plot

A new item, called Derivative, will be added to the data set in the data explorer frame, below the i vs t plot (see Figure 4.161). An additional plot, called Derivative plot is automatically added to the Derivative analysis item.

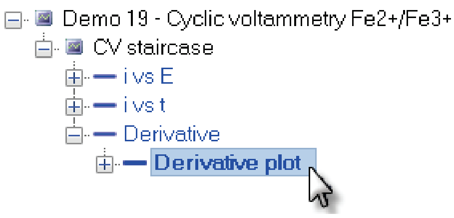


Figure 4.161 – The newly added Derivative analysis tool

Click the Derivative plot to display the first derivative of the current (see Figure 4.162).

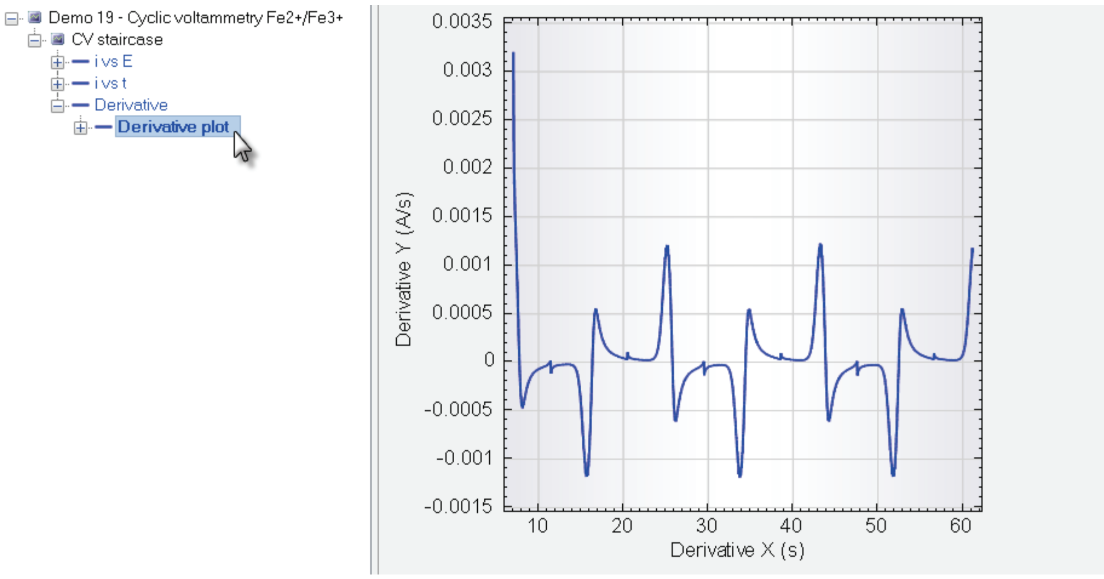


Figure 4.162 – The derivative of the i vs t plot

Note

The results of the Derivative are stored in the data grid.

4.8.1.7 – Integrate

The integrate analysis tools can be used to calculate the integral of a plot. This tool can be added to any Y vs X plot in the data analysis view.

Double click on the demo file *Demo 19 – Cyclic voltammetry Fe^{2+}/Fe^{3+}* entry of the demo database to load it into the data explorer frame. This file contains data points of a cyclic voltammetry staircase experiment recorded in a 0.05 M $[Fe(CN)_6]^{4-/3-}$ in 0.2 M NaOH solution. The number of scans is three.

Click the i vs E plot in the data explorer frame to display the data points from this demo file (see Figure 4.163). A typical reversible electron transfer behavior is recognizable in the cyclic voltammogram.

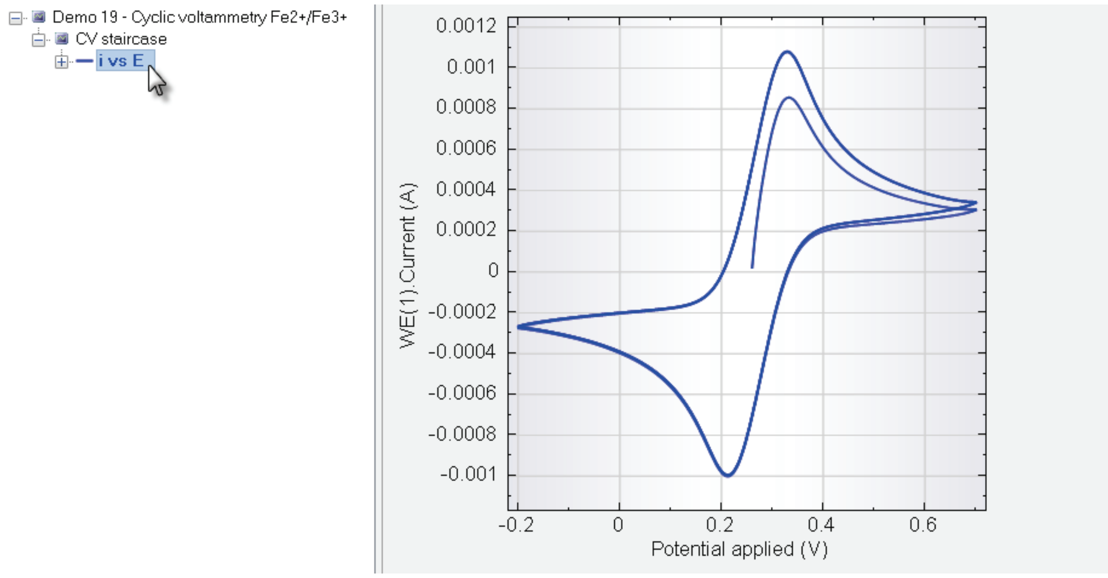


Figure 4.163 – The Demo 19 data file

Right click the CV staircase item in the data explorer frame, and select the i vs t plot from the plot context menu (see Figure 4.164).

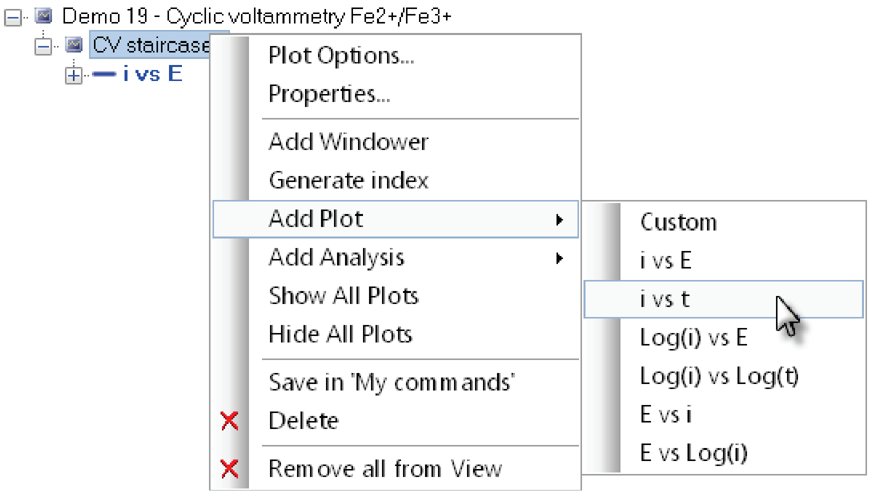


Figure 4.164 – Adding the i vs t plot to the data set

A new plot, showing the current vs time recorded during this experiment will be added to the data set. Click this new plot to display the plot in the analysis view (see Figure 4.165).

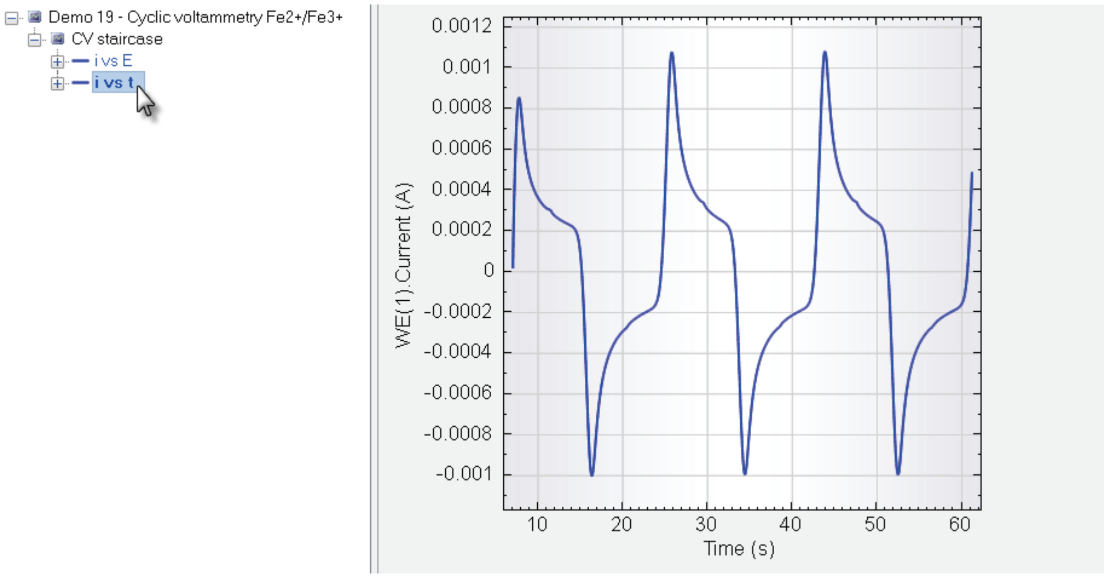


Figure 4.165 – The i vs t plot added to the data set

To calculated the integral of current vs time, right-click the i vs t item in the data explorer and select the Integrate tool from the Add Analysis menu (see Figure 4.166).

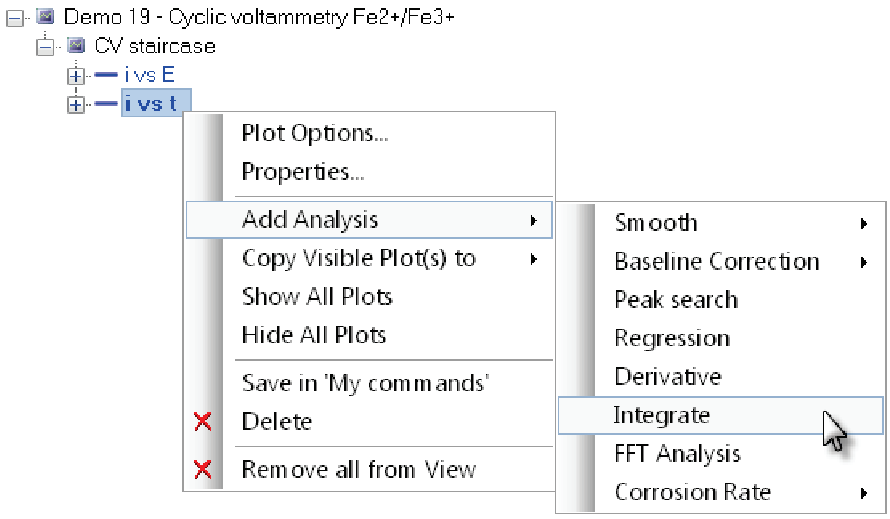


Figure 4.166 – Adding the Integrate tool to the i vs t plot

A new item, called Integrate, will be added to the data set in the data explorer frame, below the i vs t plot (see Figure 4.161).

An additional plot, called Integrate plot is automatically added to the Integrate analysis item.

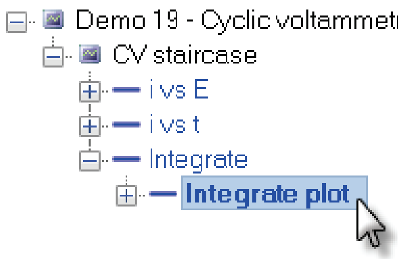


Figure 4.167 – The newly added Integrate analysis tool

Click the Integrate plot to display the integral of the current vs time (see Figure 4.168).

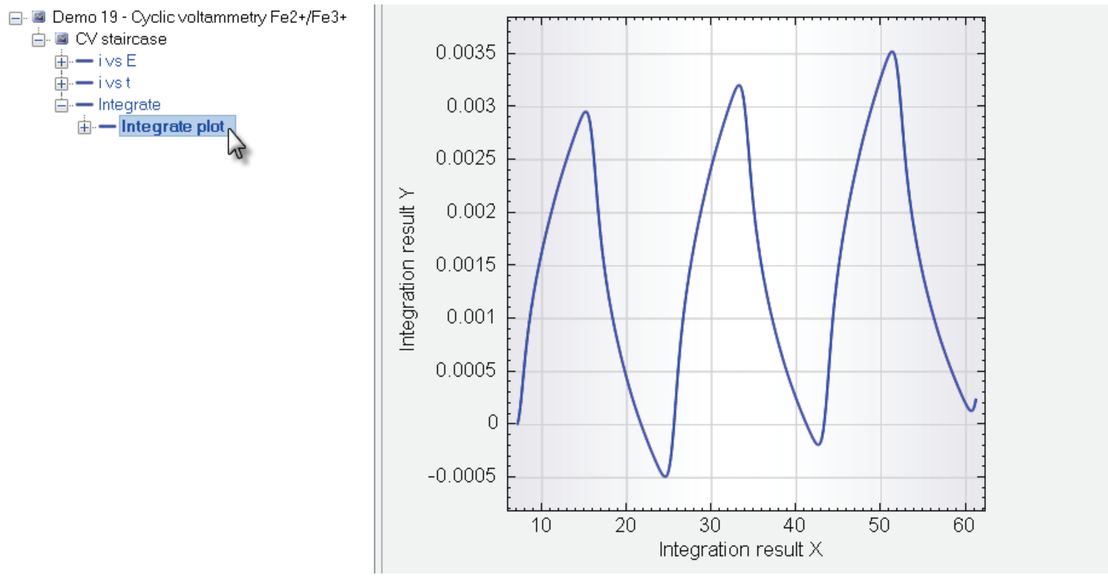


Figure 4.168 – The integral of the i vs t plot

Note

The results of the Integral are stored in the data grid.

4.8.1.8 – FFT Analysis

The FFT Analysis tool can be used to transform experimental data from the time domain into the frequency domain, in order to identify the different frequency components present in a given data set.

The transformation from time domain to frequency domain through the Fast Fourier Transform algorithm assumes that the data outside of the measured time segment is either zero or that the data in this segment repeats periodically.

Double click on the demo file *Demo 16 – FRA impedance* entry of the demo database to load it into the data explorer frame. This file contains impedance spectroscopy data points recorded on the Autolab dummy cell (c).

Click the Nyquist –Z" vs Z plot in the data explorer frame to display the data points from this demo file (see Figure 4.169). The data is presented as a Nyquist plot but it can also be plotted as a Bode plot (Bode modulus and Bode phase plots are available).

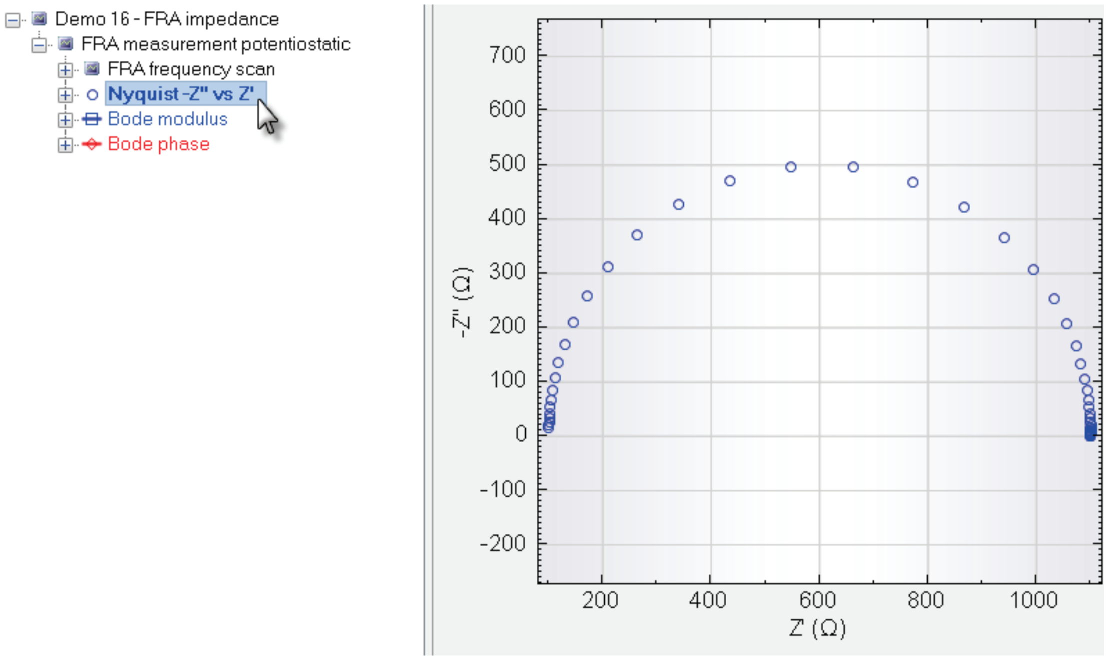


Figure 4.169 – The data from the Demo 16 data file

Note

Detailed information on impedance spectroscopy measurement can be found in the **Impedance spectroscopy tutorial**, available from the Help – Tutorials menu.

Click the ⊞ button next to the FRA frequency scan item in the data explorer. This item will be expanded and the list of the individual frequencies used during the measurement will be displayed. Expand the *#1 FRA frequency scan – 10 kHz group* and the *FRA single frequency* group to reveal three plots: E% (AC) vs t, i% (AC) vs t and Lissajous (see Figure 4.170).

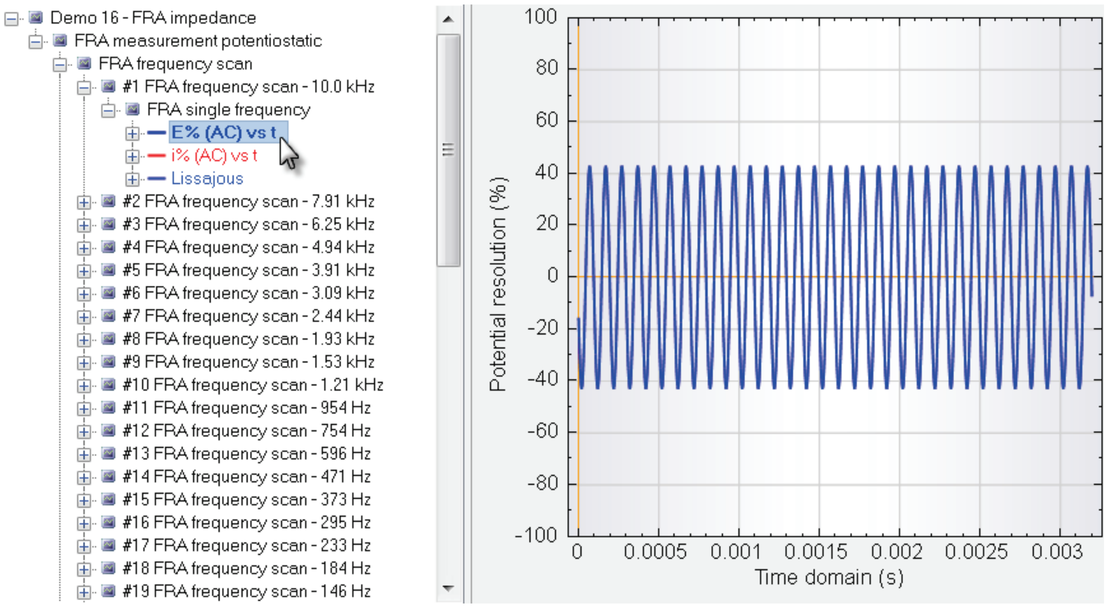


Figure 4.170 – The raw recorded values are available for each individual frequency

To use the FFT Analysis tool, right-click the E% (AC) vs t plot in the data explorer frame and, from the context menu, select the FFT Analysis tool (see Figure 4.171).

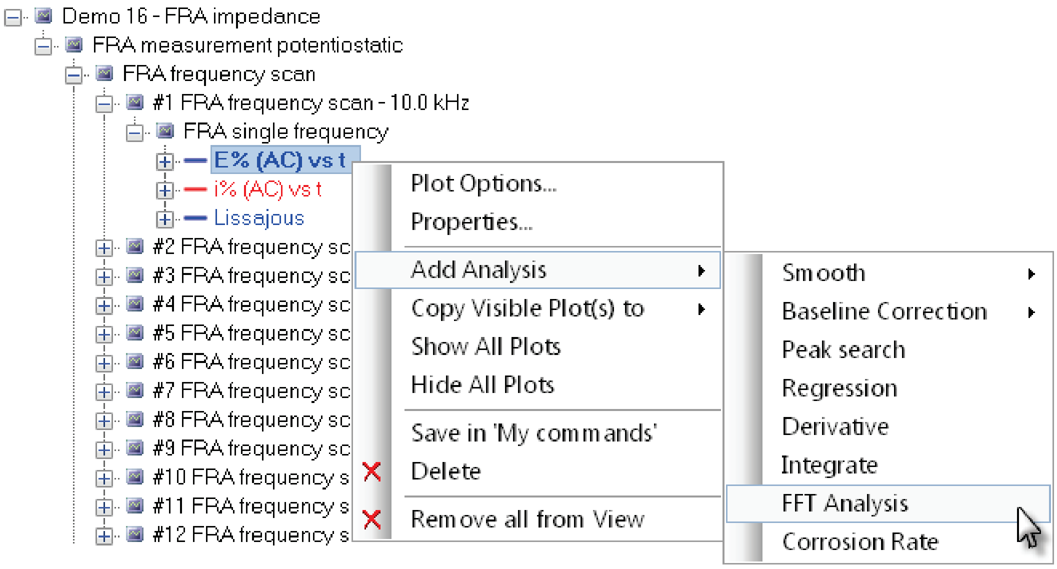


Figure 4.171 – Adding the FFT Analysis tool to the data

A new item, called FFT Analysis, will be added to the data set in the data explorer frame for the 10 kHz frequency (see Figure 4.172).

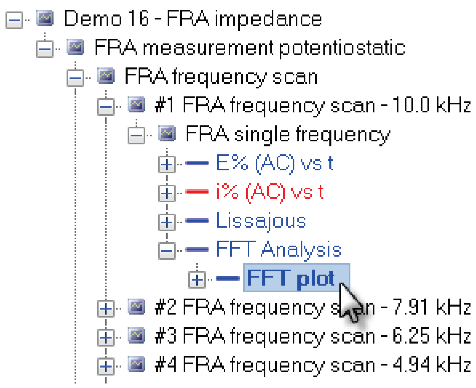


Figure 4.172 – The newly added FFT Analysis tool

Click the FFT plot generated by the FFT Analysis tool to display the transformed data (see Figure 4.173).

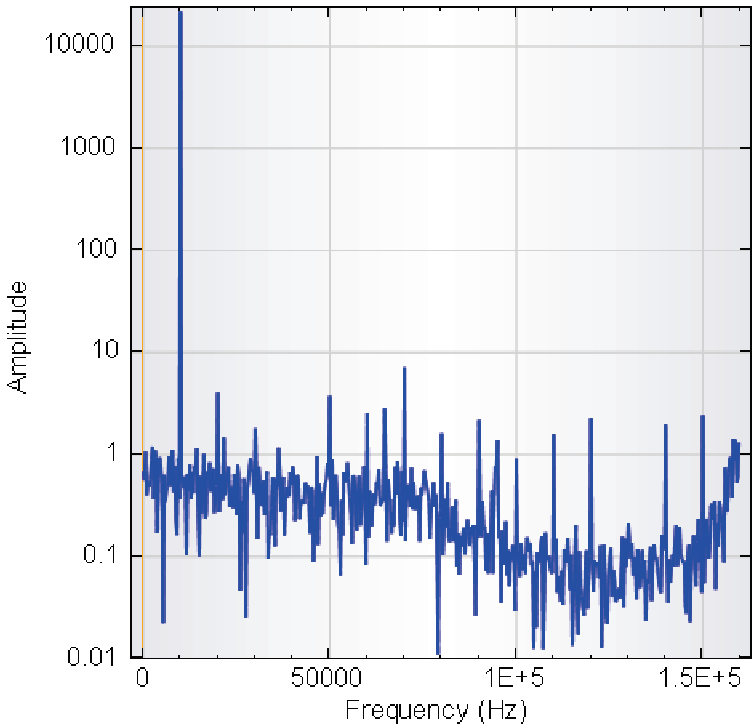


Figure 4.173 – The raw impedance data after transformation from the time domain into the frequency domain

The transformed data shows a single peak at 10 kHz consistent with the applied frequency. The rest of transformed data shows the contributions from environmental or instrumental noise.

Note

The transformed data is displayed in logarithmic scale on the Y axis. The data is shown in arbitrary units.

4.8.1.9 – Corrosion rate

The corrosion rate analysis tool can be used to convert the exchange current density in amount of material corroded per year. The corrosion rate analysis tool is typically used on a linear polarization experiments and allows the determination of the polarization resistance, the Tafel slopes and the exchange current density.

Double click on the demo file *Demo 20 – Iron screw in seawater* entry of the demo database to load it into the data explorer frame. This file contains data points of a linear sweep voltammetry experiment recorded with an iron screw in sea water[34].

Click the i vs E plot in the data explorer frame to display the data points from this demo file (see Figure 4.174).

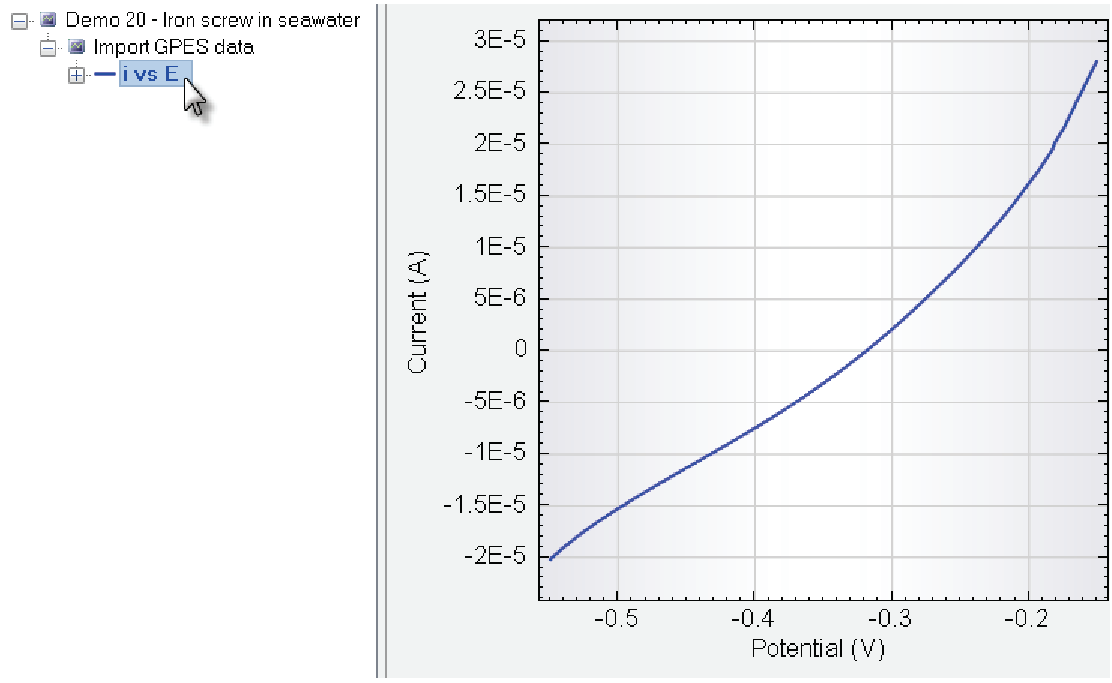


Figure 4.174 – The data provided in the Demo 20 data file

To use the corrosion rate analysis tool on this data set, right-click the i vs E item in the data explorer frame and select the Corrosion Rate from the Add analysis context menu (see Figure 4.175).

[34] This data set is an imported GPES file.

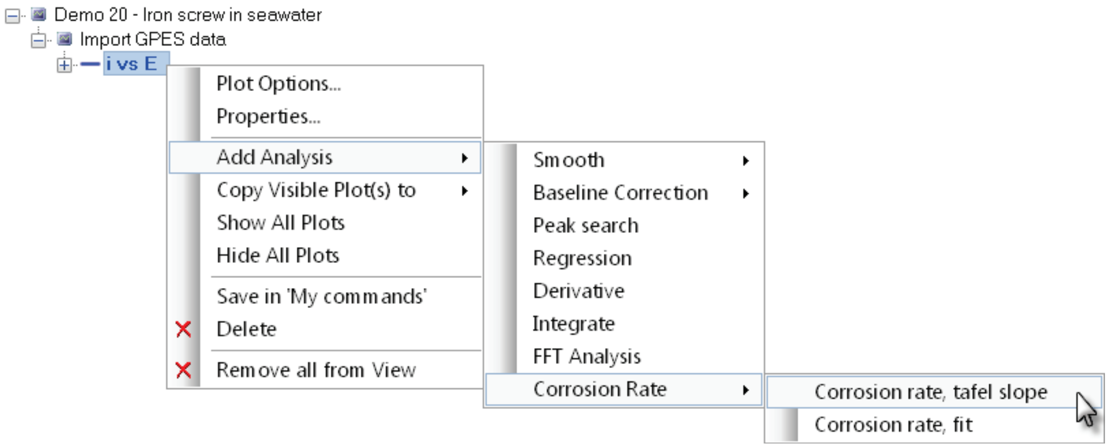


Figure 4.175 – The Corrosion Rate analysis tool can be added to the data through the right-click menu

Two different corrosion rate tools are available from the context menu:

- **Corrosion rate, Tafel slope:** this tool is used to calculate the Tafel slope and the determination of the exchange current density.
- **Corrosion rate, fit:** this tool can be used to perform a calculation similar to that of the Tafel slope tool. Additionally, the data is fitted using the Butler-Volmer equation:

$$i = i_{corr}\left[e^{2.303\frac{E-E_{corr}}{b_a}} - e^{2.303\frac{E-E_{corr}}{b_c}}\right]$$

Where i_{corr} is the corrosion current, E_{corr} is the corrosion potential, b_a and b_c are the anodic and cathodic Tafel constants, respectively.

4.8.1.9.1 – Corrosion rate, Tafel slope

When the Tafel slope analysis tool is added to the data as described in the previous section (see Figure 4.175), a new item is added to the data explorer frame.

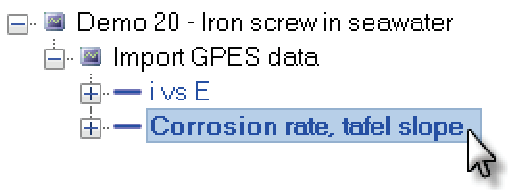


Figure 4.176 – The corrosion rate, Tafel slope is added to the data set

Click the corrosion rate data analysis item to display the data from the data set, in logarithmic scaling (see Figure 4.177).

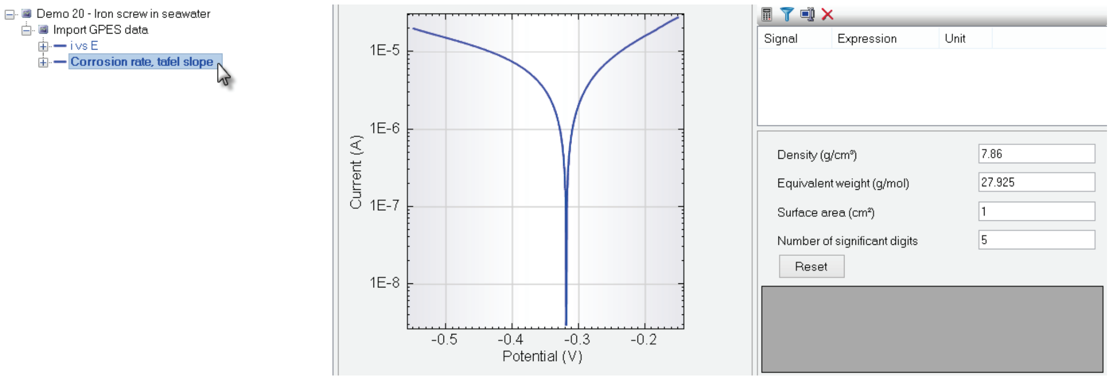


Figure 4.177 – The Tafel slope analysis tool automatically displays the experimental data in logarithmic scaling

Additionally, the control parameters of the Tafel slope analysis tool will be displayed on the right-hand side of the plot. This part of the view allows the definition of parameter for the Tafel slope analysis (see Figure 4.178).

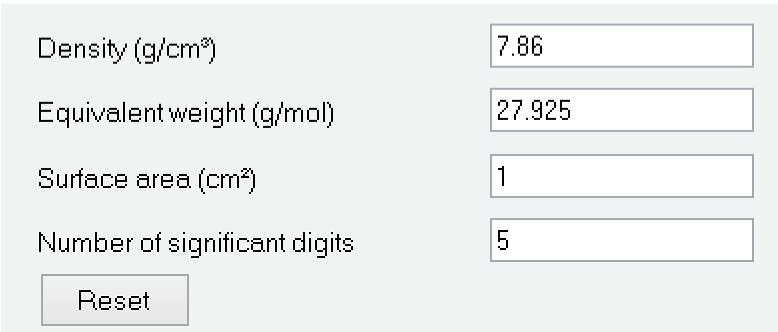


Figure 4.178 – The parameters for the Tafel slope analysis

The following parameters are available:

- **Density:** specifies the density of the sample in g/cm^3
- **Equivalent weight:** defines the equivalent weight of the sample in g/mol of exchanged electrons
- **Surface area:** defines the area of the sample, in cm^2
- **Number of significant digits:** Defines the number of significant digits used in the Analysis results frame.

For high cathodic or anodic over potentials, the Butler-Volmer equation can be reduced to the following linear expressions, respectively:

$$E - E_{corr} = \log i_{corr} + b_a \log|i|$$

$$E - E_{corr} = \log i_{corr} - b_c \log|i|$$

When the Tafel slope analysis tool is selected, the mouse pointer changes to a cross. Using the mouse pointer, click two points in the plot to specify the linear part of the anodic branch (see Figure 4.179). Once the second point is clicked, a line will be drawn on the plot.

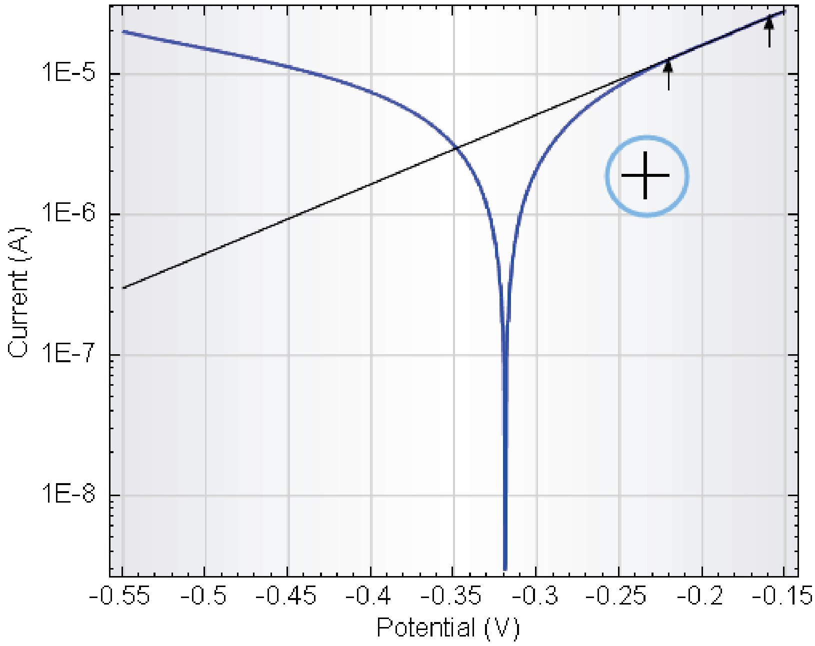


Figure 4.179 – The Tafel slope analysis tools requires the specification of the linear segments of the Tafel plot (1/2)

The same must be repeated for the cathodic branch of the plot. Once both linear segments have been defined, the intercept is determined and indicated in the plot (see Figure 4.180).

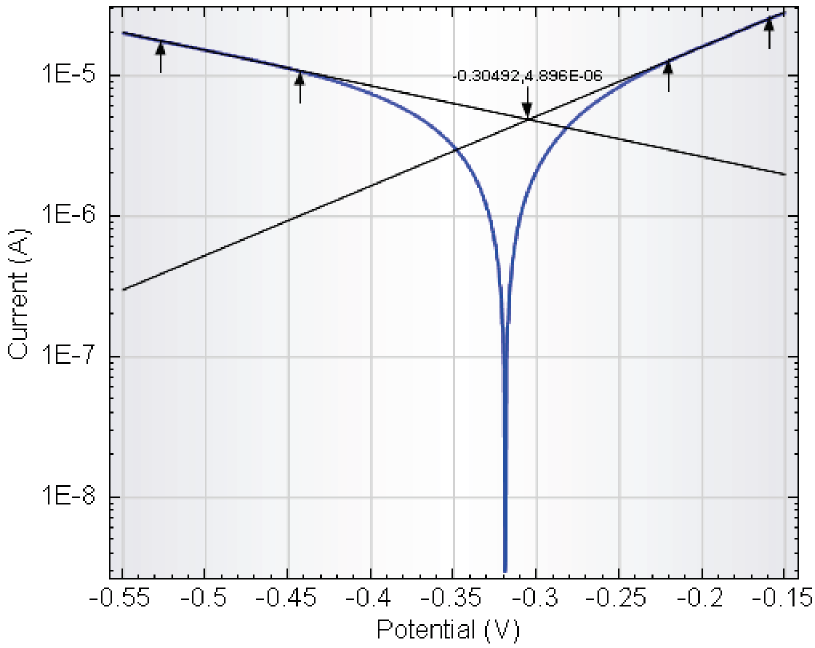


Figure 4.180 - The Tafel slope analysis tools requires the specification of the linear segments of the Tafel plot (2/2)

Note

Select the anodic branch of the plot first and then the cathodic branch of the plot.

The intercept on the Tafel plot provides an estimation of the corrosion current and the corrosion potential. These values are labeled on the plot. The complete details of the calculation are summarized in the data explorer frame (see Figure 4.181).

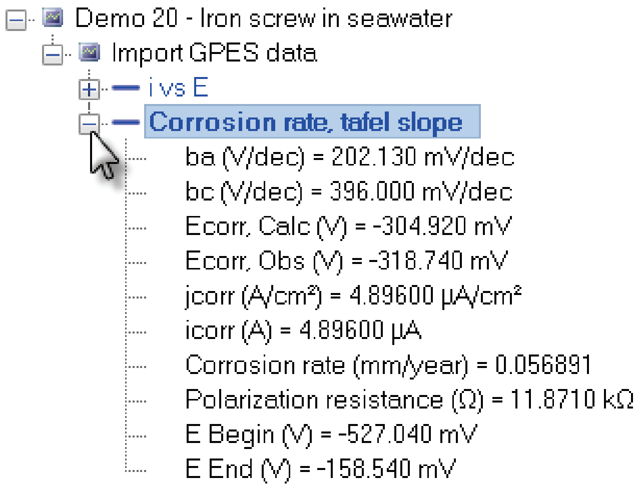


Figure 4.181 – The results of the Tafel slope analysis are shown in the data explorer frame

The following information is provided in the data explorer frame:

- **b_a & b_c:** the anodic and cathodic Tafel coefficient, in V/decade.
- **Ecorr, Calc:** the corrosion potential calculated from the intercept on the Tafel plot, in Volts.
- **Ecorr, Obs:** the observed experimental corrosion potential, in Volts.
- **jcorr and icorr:** the exchange current density and exchange current calculated from the intercept on the Tafel plot, in A/cm^2 and A, respectively.
- **Corrosion rate:** the estimated corrosion rate of the material, calculated from the intercept on the Tafel plot, in mm/year.
- **Polarization resistance:** the estimated value of the polarization resistance, calculated from the intercept and the Tafel slopes, according to:

$$R_p = \frac{1}{2.303\left(\frac{1}{b_a} + \frac{1}{b_c}\right) i_{corr}}$$

- **E Begin, E End:** the voltage limits corresponding to the measurement.

4.8.1.9.2 – Corrosion rate, Fit

The Tafel slope analysis tool provides an estimation of the polarization resistance and the exchange current density. Accurate determination of these parameters can be done by fitting the Butler-Volmer equation to the experimental data set, taking into account that the corrosion current is related to the polarization resistance and the Tafel slopes, according to:

$$\frac{1}{i_{corr}} = R_p \cdot 2.303 \left(\frac{1}{b_a} + \frac{1}{b_c}\right)$$

This expression can be used in combination with the Butler-Volmer equation in order to perform a non-linear fit of the equation to the data set. In order to do this in NOVA, the Corrosion rate, fit analysis tool can be used. To add this analysis tool to a data set, right-click the i vs E plot, and select the Corrosion rate, fit analysis tool from the Add Analysis menu (see Figure 4.182).

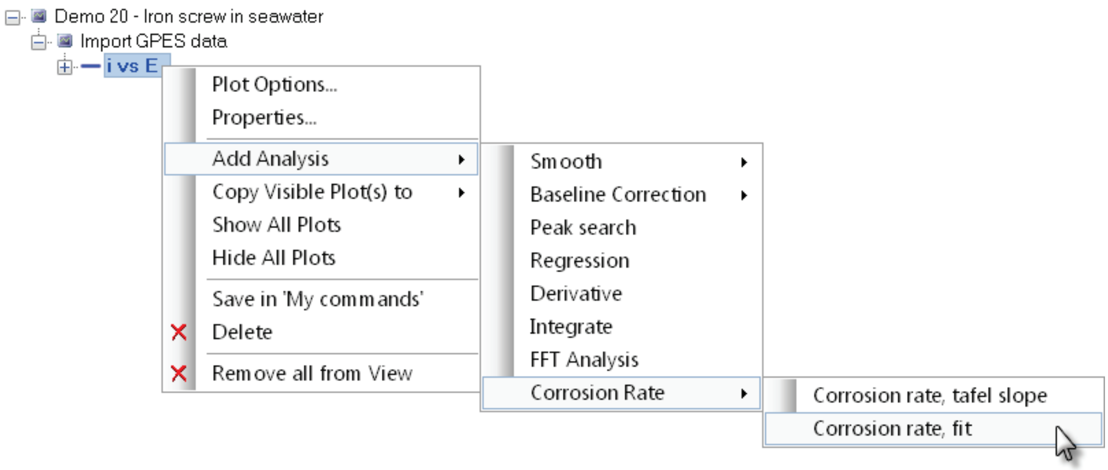


Figure 4.182 – Adding the corrosion rate, fit analysis tool to the data set

A new item, called Corrosion rate, fit will be added to the data set in the data explorer frame (see Figure 4.183).

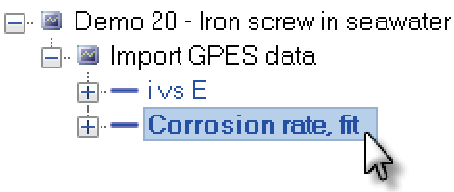


Figure 4.183 – The corrosion rate, fit is added to the data set

Click the corrosion rate data analysis item to display the data from the data set, in logarithmic scaling (see Figure 4.184).

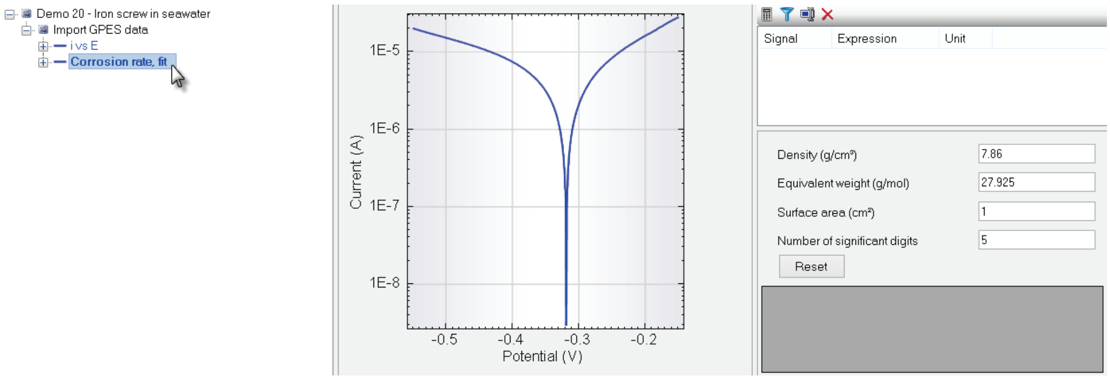


Figure 4.184 – The corrosion rate, fit analysis tool automatically displays the experimental data in logarithmic scaling

Additionally, the control parameters of the corrosion rate, fit analysis tool will be displayed on the right-hand side of the plot. This part of the view allows the definition of parameter for the corrosion rate analysis, similarly to the Tafel slope analysis item (see Figure 4.185).

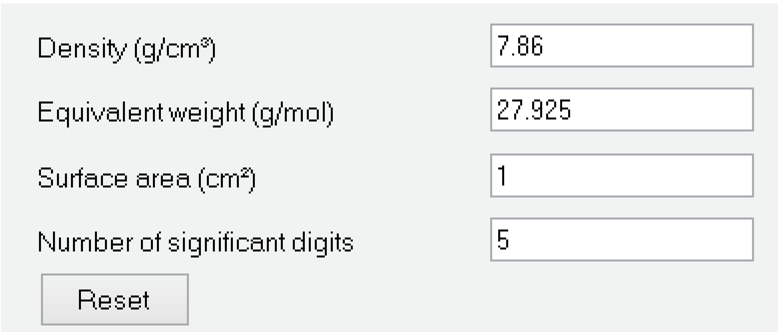


Figure 4.185 – The parameters for the corrosion rate, fit analysis tool

The following parameters are available:

- **Density:** specifies the density of the sample in g/cm^3
- **Equivalent weight:** defines the equivalent weight of the sample in g/mol of exchanged electrons
- **Surface area:** defines the area of the sample, in cm^2
- **Number of significant digits:** Defines the number of significant digits used in the Analysis results frame.

When the corrosion rate, fit analysis tool is selected, the mouse pointer changes to a cross. Using the mouse pointer, click two points in the plot to specify the linear part of the anodic branch (see Figure 4.186). Once the second point is clicked, a line will be drawn on the plot.

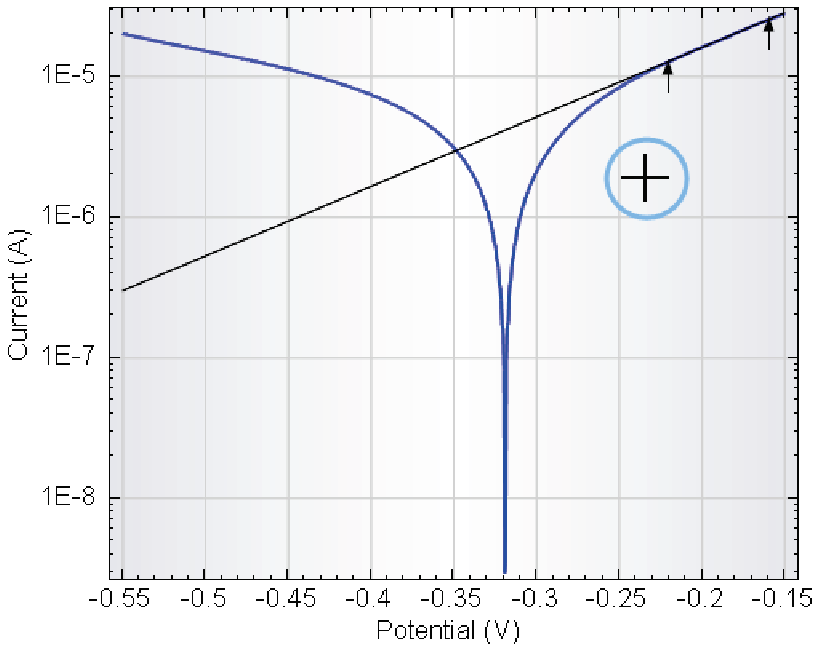


Figure 4.186 – The corrosion rate, fit analysis tools requires the specification of the linear segments of the Tafel plot (1/2)

The same must be repeated for the cathodic branch of the plot. Once both linear segments have been defined, the intercept is determined and indicated in the plot (see Figure 4.187).

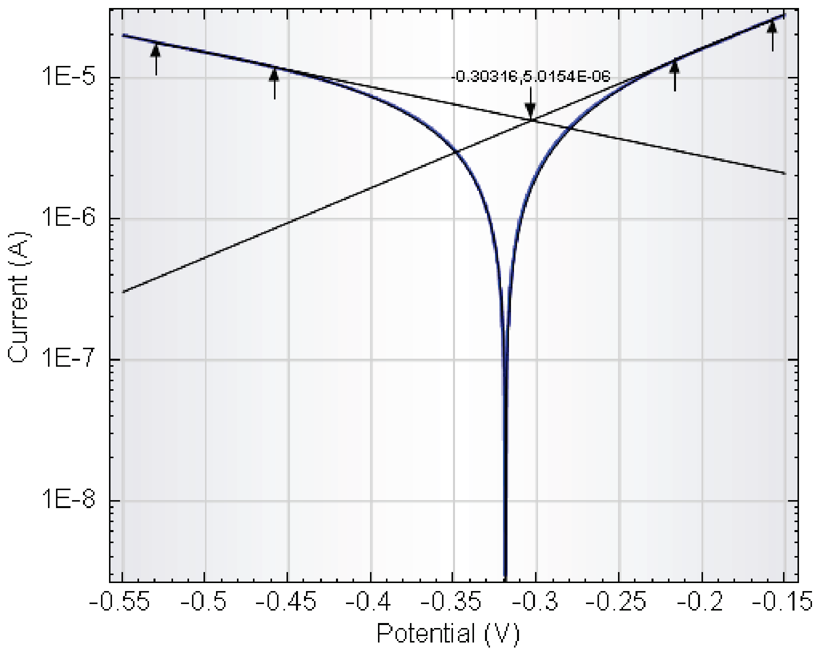


Figure 4.187 - The corrosion rate, fit analysis tools requires the specification of the linear segments of the Tafel plot (2/2)

Note

Select the anodic branch of the plot first and then the cathodic branch of the plot.

The intercept on the Tafel plot provides an estimation of the corrosion current and the corrosion potential. These values are used as initial conditions for the fitting algorithm. After convergence has been achieved, the complete details of the calculation are summarized in the data explorer frame (see Figure 4.188).

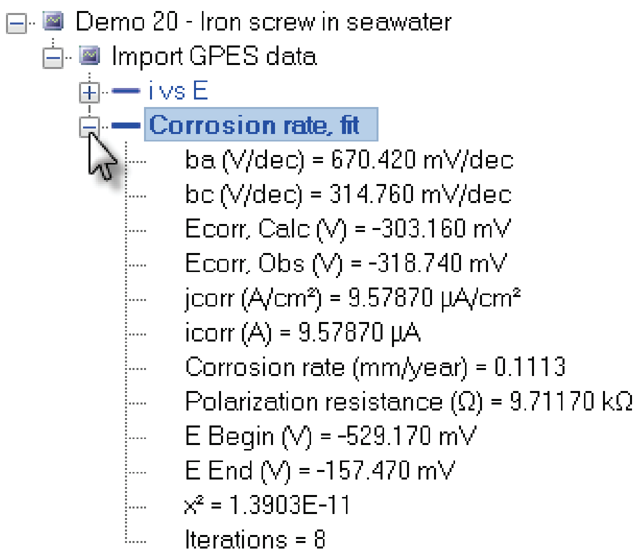


Figure 4.188 – The results of the Tafel slope analysis are shown in the data explorer frame

The reliability of the fitting results greatly depends on the data provided as input for the fitting algorithm. A number of conditions must be verified for the data points to be valid[35]. Particular care must be taken when defining the linear section of the Tafel plot at large overpotentials. As a rule of thumb, the linearity of the Tafel plot should extend over at least one decade of current.

The following information is provided in the data explorer frame:

- **b_a & b_c:** the anodic and cathodic Tafel coefficient, in V/decade.
- **Ecorr, Calc:** the corrosion potential calculated from the intercept on the Tafel plot, in Volts.
- **Ecorr, Obs:** the observed experimental corrosion potential, in Volts.

[35] See A. J. Bard and L. R. Faulkner, Electrochemical Methods, Fundamentals and Application, 2nd Edition, Wiley (NY), Chapter 3; R. Baboian, Electrochemical Techniques for Corrosion Engineering, NACE; C. H. A. Brett and A. M. O. Brett, Electrochemistry, Principles, Methods and Applications, Oxford Science Publications; D. C. Silverman, Practical Corrosion Prediction Using Electrochemical Techniques, Uhlig's Corrosion Handbook, 2nd Edition.

- **jcorr and icorr:** the exchange current density and exchange current calculated from fitting of the Butler-Volmer equation to the experimental data, in A/cm^2 and A, respectively.
- **Corrosion rate:** the estimated corrosion rate of the material, calculated from the intercept on the Tafel plot, in mm/year.
- **Polarization resistance:** the estimated value of the polarization resistance, calculated from fitting algorithm.
- **E Begin, E End:** the voltage limits corresponding to the measurement.
- **χ^2:** the squared sum of the difference between the data points of the fit and the experimental data points.
- **Iterations:** the number of iteration used during the fitting of the data.

Note

The results of both the Tafel slope analysis and the corrosion rate analysis are stored in the data grid.

4.8.2 – Data set analysis tools

The data set analysis tools can be added to a complete data set in the analysis view, using the right-click menu (see Figure 4.189). These analysis tools differ from those that fall into the X/Y analysis tools category because they require more information that a set of X and Y values. Therefore, these tools are added to a complete data set and not to a plot. This section provides an overview of the available analysis commands in this category.

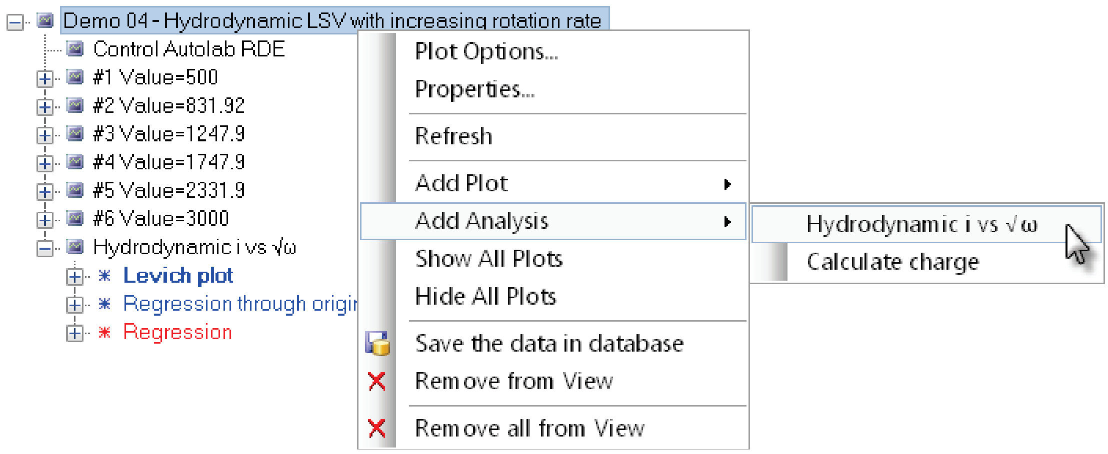


Figure 4.189 – Data set analysis tools can be added directly to a plot in the analysis view

The example shown in Figure 4.189 corresponds to the Hydrodynamic i vs $\sqrt{\omega}$ analysis tool. This tool is used to create a Levich plot, using the limiting current values recorded at different rotation rates. Two linear regressions are automatically included.

4.8.2.1 – iR drop correction

This data analysis tool can be used to correct the measured data for the voltage drop introduced by the uncompensated resistance. This tool is used to correct the measured for ohmic drop. This tool requires a value of the uncompensated resistance to be specified, in Ohm. This value is used to calculate a new potential scale, according to:

$$E_{calculated} = i \cdot R_u$$

where i is the measured current (WE(1).Current) and R_u is the specified uncompensated resistance.

Double click on the demo file *Demo 19 – Cyclic voltammetry Fe^{2+}/Fe^{3+}* entry of the demo database to load it into the data explorer frame. This file contains data points of a cyclic voltammetry staircase experiment recorded in a 0.05 M $[Fe(CN)_6]^{4-/3-}$ in 0.2 M NaOH solution. The number of scans is three.

To add the iR drop correction analysis tool to this data set, right-click the CV staircase item in the data explorer frame and select the iR drop correction analysis item from the context menu (see Figure 4.190).

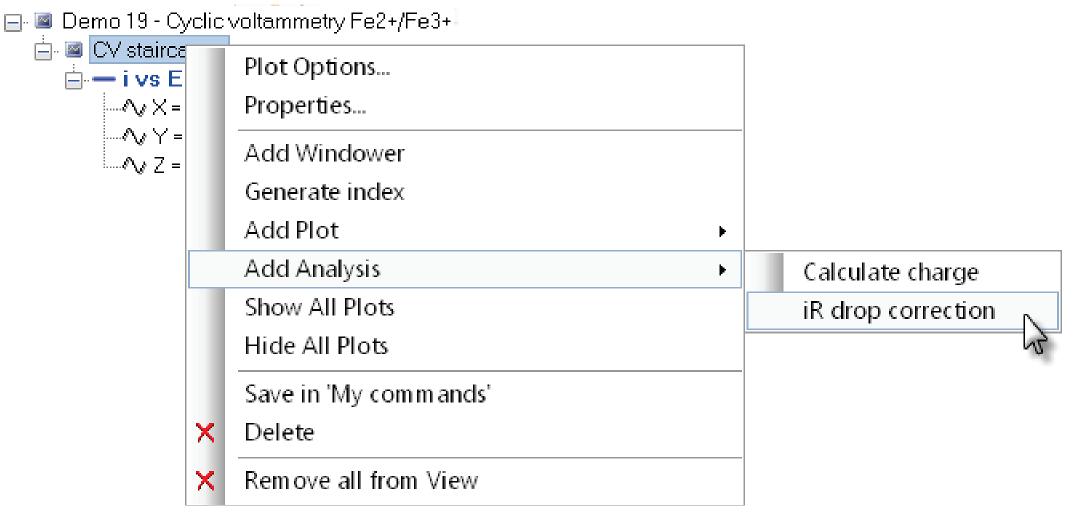


Figure 4.190 – Select the iR drop correction analysis tool to add it to the data set

A popup window will be displayed. The uncompensated resistance can be specified in the window, in Ohm (see Figure 4.191).

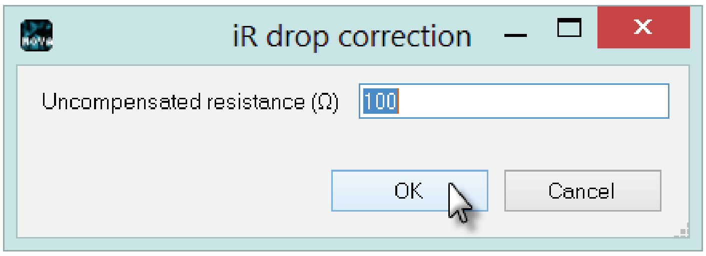


Figure 4.191 – The uncompensated resistance value can be specified in a popup window

A new item, called iR drop correction, will be added to the data set, below the CV staircase item. A new signal, called Corrected potential, will be added to the dataset. This new signal can be used to create or change a plot and use the Corrected potential (see Figure 4.192).

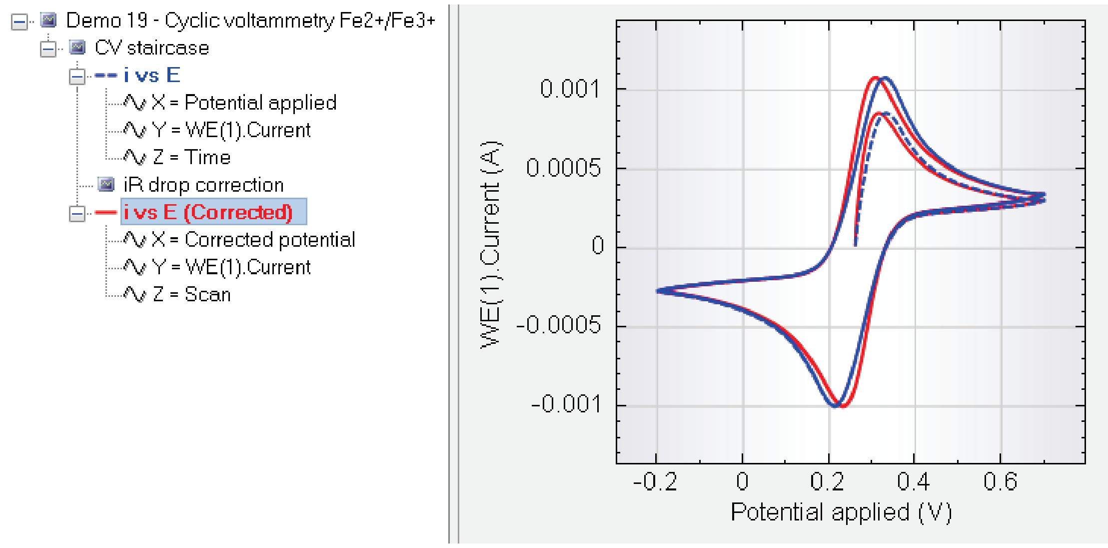


Figure 4.192 – Using the corrected potential scale (overlay of source data and corrected data)

It is possible to change the value of the uncompensated resistance by clicking the iR drop correction item in the data explorer. The input field for the Uncompensated resistance will be displayed on the right-hand side of the plot (see Figure 4.193).

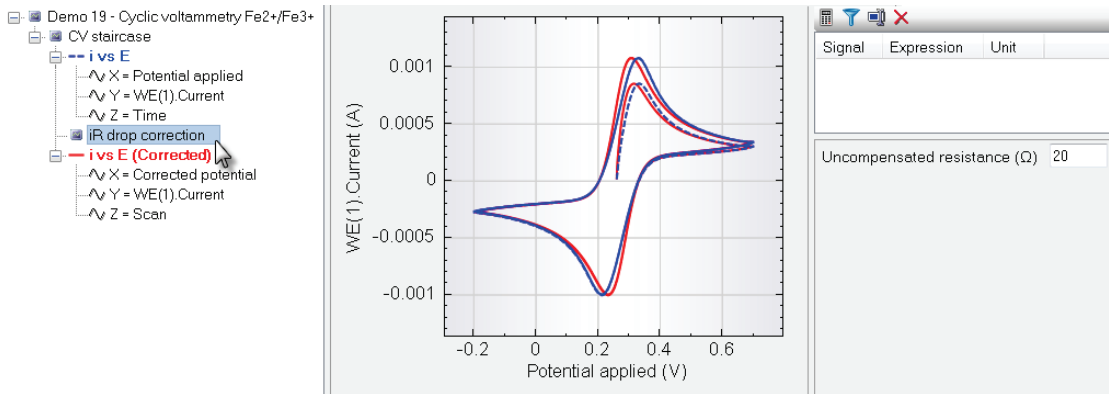


Figure 4.193 – The uncompensated resistance can be adjusted at any time by clicking the iR drop correction item in the data explorer

4.8.2.2 – Hydrodynamic i vs $\sqrt{\omega}$

This data analysis tool can be used to create a Levich plot, using the limiting current values recorded in an experiment using forced convection by means of the Autolab rotating disc electrode.

Typically, in this type of experiment, a potential scan is performed at different rotation rates. As the rotation rate increases, the convective drag from the electrode also increases and the diffusion layer decreases in thickness. As a result, the current values in the mass-transport controlled region increase.

Under these experimental conditions, the limiting current values, i_l are related to the rotation rate of the working electrode according to:

$$i_l = 0.62 \cdot AnFD^{\frac{2}{3}}\nu^{-\frac{1}{6}}C^{\infty}\sqrt{\omega}$$

Where A is the geometric area of the working electrode, n is the number of electrons involved in the electrochemical reaction, F is the Faraday constant, D is the diffusion coefficient, ν is the kinematic viscosity, C^{∞} is the bulk concentration of the electroactive species and ω is the angular frequency of the working electrode.

Double click on the demo file *Demo 04 - Hydrodynamic LSV with increasing rotation rate* entry of the demo database to load it into the data explorer frame. This file contains data points of six linear sweep voltammetry experiment recorded in a $[Fe(CN)_6]^{4-/3-}$ 0.05 M in 0.2 M NaOH solution at different rotation rates. A 3 mm platinum disc was used as working electrode.

Note

This example was already used in Section 4.8.1.5.

Figure 4.194 shows an overlay of the linear sweep voltammogram recorded at different rotation rates.

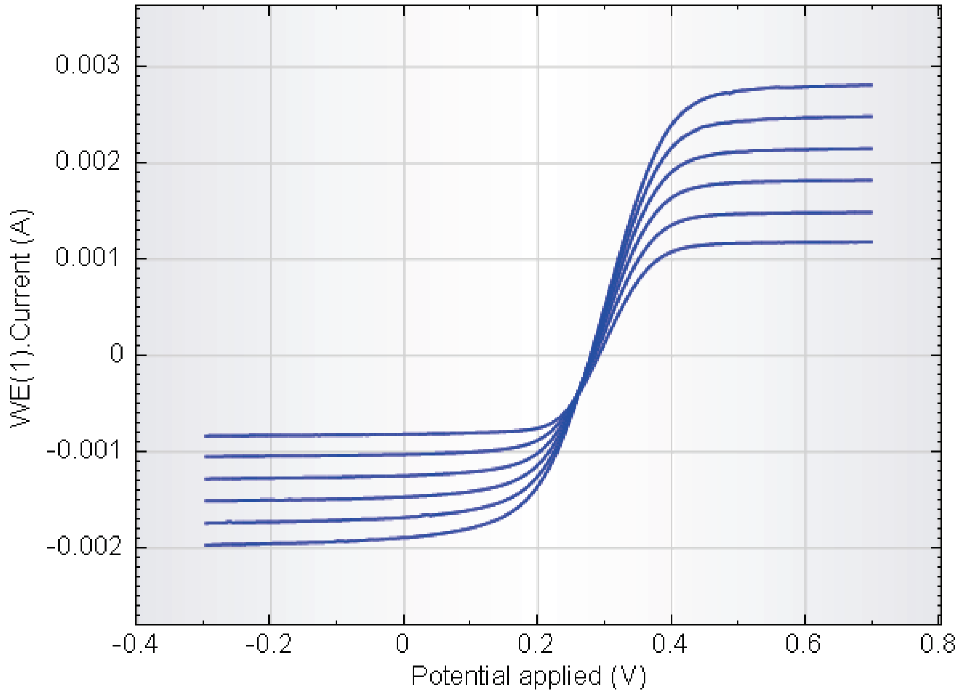


Figure 4.194 – The linear sweep voltammograms recorded at different rotation rates

This data set was recorded using the default Autolab Hydrodynamic linear sweep procedure. This procedure automatically adds the Hydrodynamic i vs $\sqrt{\omega}$ analysis tool to the data at the end of the experiment. This tool is therefore already added to the data (see Figure 4.195).

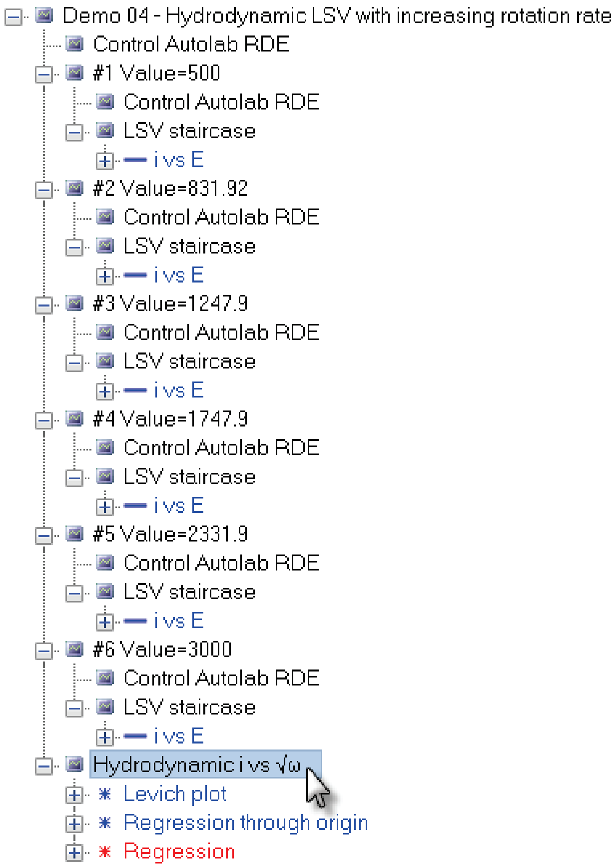


Figure 4.195 – The Hydrodynamic i vs √ω analysis tool is already added to the data

For illustration purposes, delete the Hydrodynamic i vs $\sqrt{\omega}$ analysis tool added to this data set by selecting this item in the data explorer frame and pressing the Delete key on the keyboard. It is also possible to right-click this item and select the delete option from the context menu (see Figure 4.196).

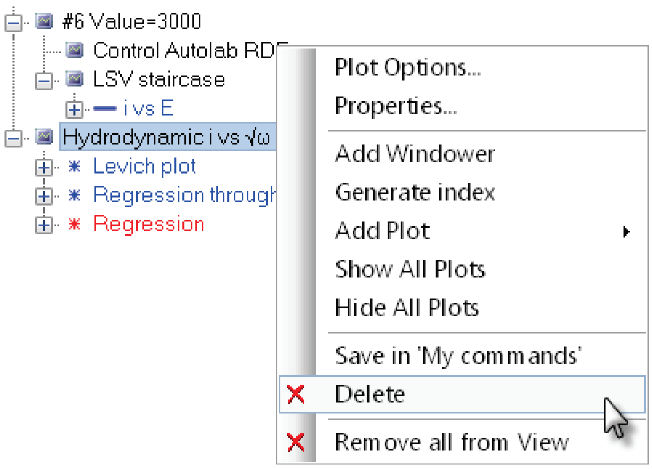


Figure 4.196 – Right-click the analysis item and select the Delete option from the context menu to remove the data analysis tool from the data set

Once the item has been removed, right-click the data set header in the data explorer frame and select the Hydrodynamic i vs $\sqrt{\omega}$ analysis tool from the Add analysis context menu (see Figure 4.197).

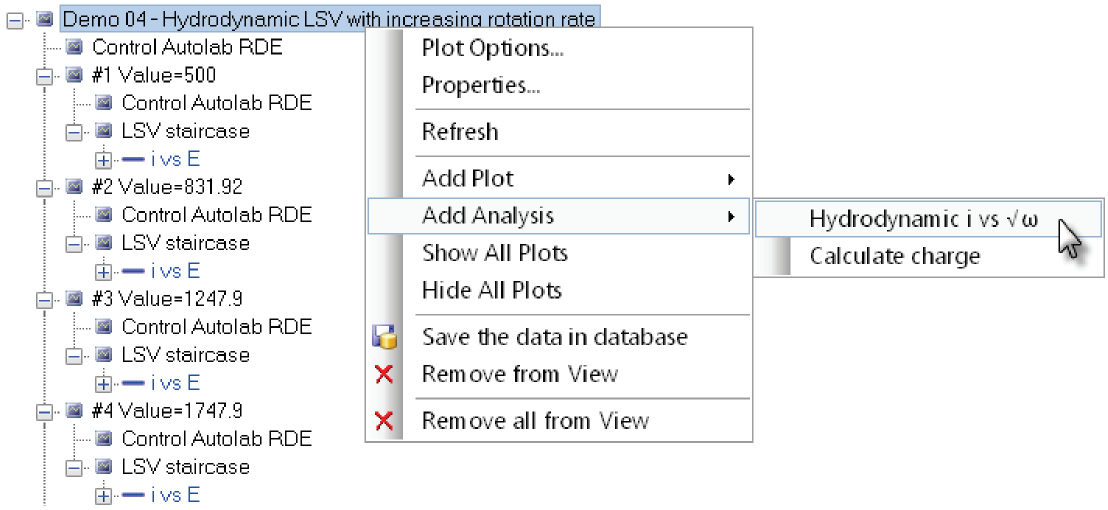


Figure 4.197 – Adding the Hydrodynamic i vs $\sqrt{\omega}$ analysis tool can be done through the right-click menu

The Hydrodynamic i vs $\sqrt{\omega}$ analysis tool will be added again to the data set, providing a Levich plot as well as two different linear regression lines (see Figure 4.198).

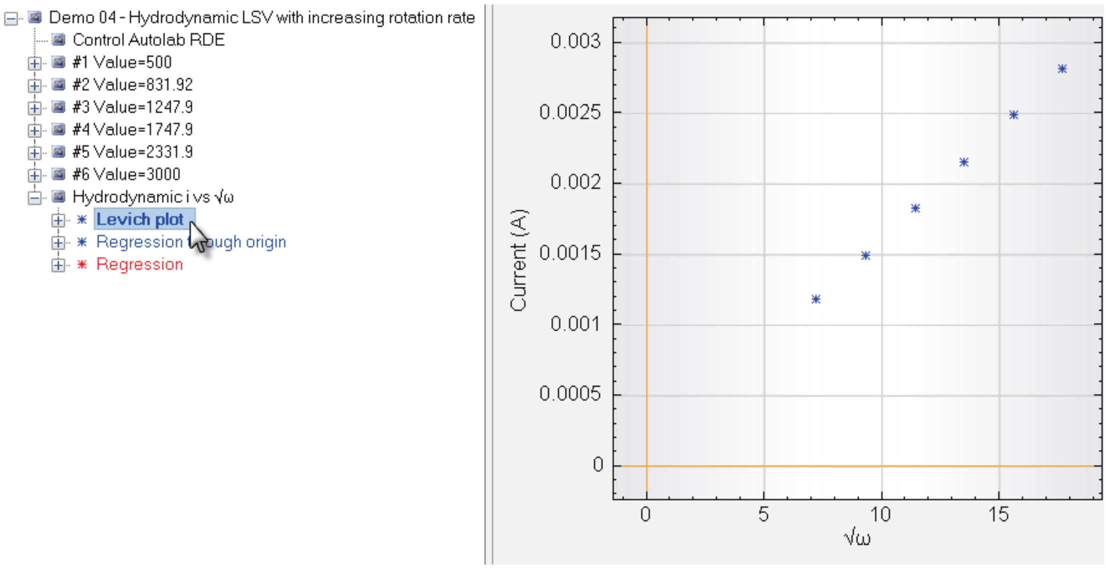


Figure 4.198 – The Hydrodynamic i vs $\sqrt{\omega}$ analysis tool generates the Levich plot automatically

Note

The Levich plot is always built using the first measured data point for each rotation rate.

4.8.2.3 – Calculate charge

This data analysis tool can be used to calculate the charge involved in an electrochemical measurement. This analysis tool can be added to a data set using the right-click menu. The result of this analysis tool is different from the result of the Integrate data analysis tool shown in Section 4.8.1.7. The integrate tool integrates the Y signal versus the X signal, regardless of the type of signal plotted. The calculate charge analysis tool calculates the charge in coulombs by integrating the current versus the time.

Double click on the demo file *Demo 19 – Cyclic voltammetry Fe^{2+}/Fe^{3+}* entry of the demo database to load it into the data explorer frame. This file contains data points of a cyclic voltammetry staircase experiment recorded in a 0.05 M $[Fe(CN)_6]^{4-/3-}$ in 0.2 M NaOH solution. The number of scans is three.

To add the Calculate charge analysis tool to this data set, right-click the CV staircase item in the data explorer frame and select the Calculate charge analysis item from the context menu (see Figure 4.199).

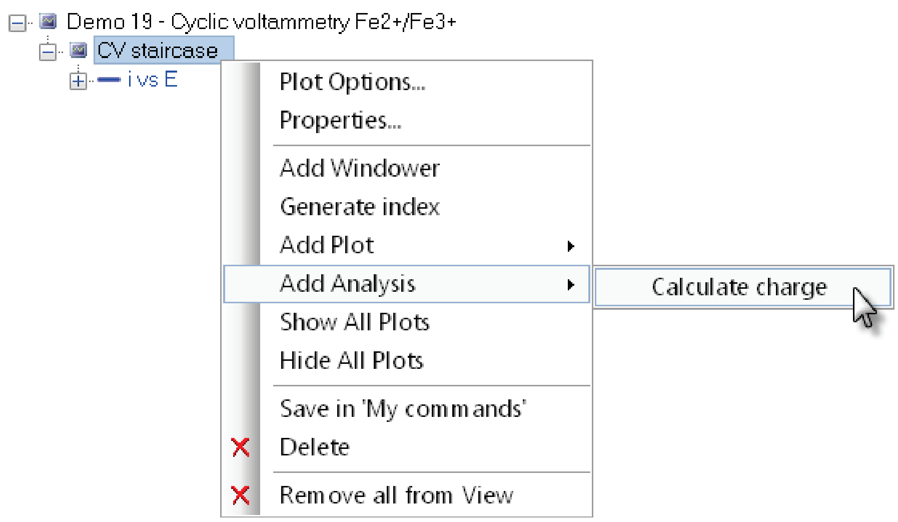


Figure 4.199 – Select the Calculate charge analysis tool to add it to the data set

A new item, called Calculate charge, will be added to the data set, below the CV staircase item. Two plots, Q vs E and Q vs t are automatically generated by this analysis tool (see Figure 4.200).

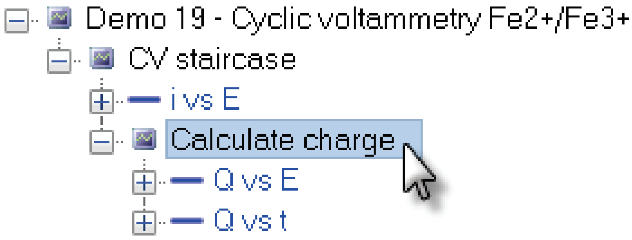


Figure 4.200 – The Calculate charge analysis item automatically generates the Q vs E and the Q vs t plots

Click the Q vs t plot to display the plot. The plot should be similar to the plot obtained with the same data set in combination with the integrate analysis tool (see Figure 4.201 and Figure 4.168).

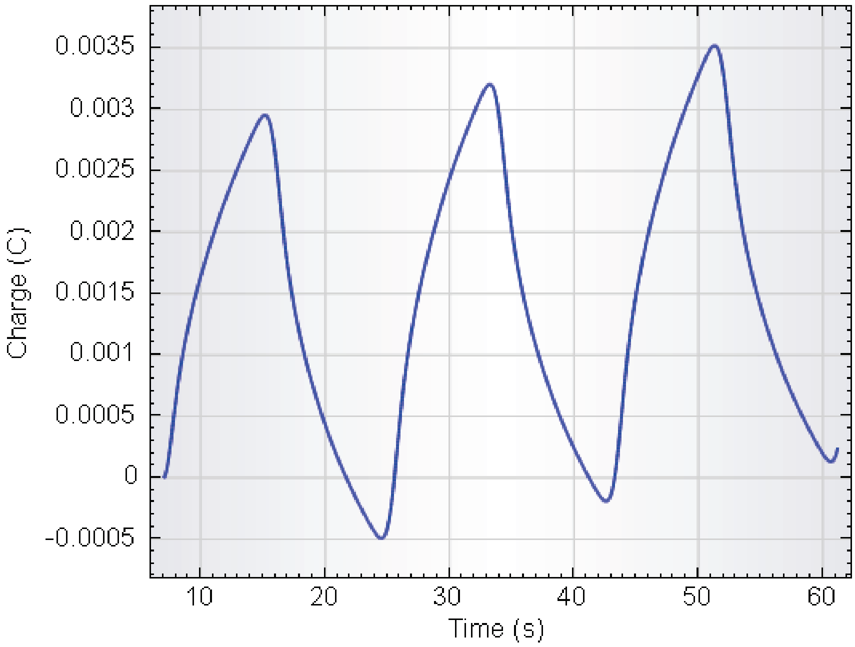


Figure 4.201 – The Q vs t plot generated by the calculate charge analysis tool

Click the Q vs E plot to display the same information plotted versus the applied potential. The plot should look like the one shown in Figure 4.202.

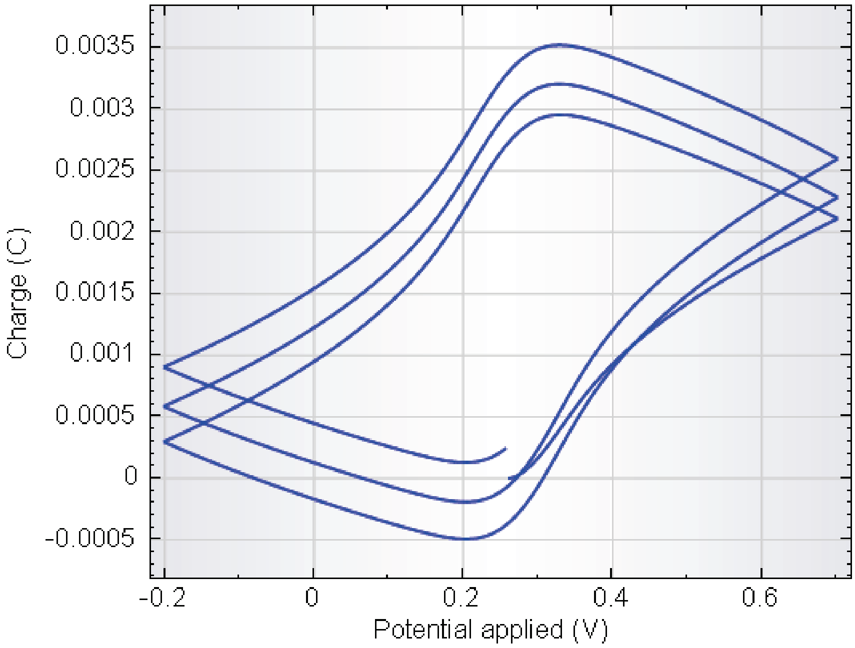


Figure 4.202 – The Q vs E plot generated by the calculate charge analysis tool

> **Note**
>
> The data provided by the Calculate charge command is stored in the data grid.

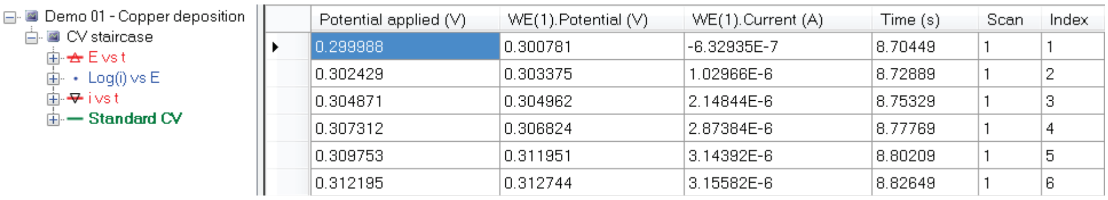
4.9 – The data grid

The data grid works as a spreadsheet that can be used to perform calculations on measured signals, to create new signals, to filter experimental data points or procedure parameters, and to export the data to Excel or ASCII. New signals created in the grid can then be used to plot the measured data in a different way.

To access the data grid, click the corresponding button in the data analysis toolbar (see Figure 4.203).

Figure 4.203 – Clicking the Show data grid button in the toolbar will display the data grid instead of a 2D or 3D plot

The data grid is shown instead of a 2D or a 3D plot and appears as a series of columns containing the measured values of each signal. In the case of the cyclic voltammogram of example #1, the displayed values correspond to WE(1).Potential applied, WE(1).Current, Time and WE(1).Potential, Index, Scan (see Figure 4.204).



	Potential applied (V)	WE(1).Potential (V)	WE(1).Current (A)	Time (s)	Scan	Index
▸	0.299988	0.300781	-6.32935E-7	8.70449	1	1
	0.302429	0.303375	1.02966E-6	8.72889	1	2
	0.304871	0.304962	2.14844E-6	8.75329	1	3
	0.307312	0.306824	2.87384E-6	8.77769	1	4
	0.309753	0.311951	3.14392E-6	8.80209	1	5
	0.312195	0.312744	3.15582E-6	8.82649	1	6

Figure 4.204 – Displaying the data grid of the data set from example #1

You can scroll down to inspect every value of these signals. For each value of WE(1).Potential applied, a corresponding value of WE(1).Current, Time, WE(1).Potential, index and scan is displayed.

Note

The data grid shows the values for the whole data set which means that the highlighted plot has no influence on the displayed. Each plot uses the same set of data points.

Note

The data grid shown in Figure 4.204 displays two columns containing values of the potential. The first one is labeled **Potential applied** while the other is labeled **WE(1).Potential**. The Potential applied signal contains the values of the potential set by the software during the measurement, while the WE(1).Potential signal shows the values of the potential recorded during the experiment.

Using the data grid, it is possible to export the measured data points to other software packages for data analysis (Excel, Origin, SigmaPlot, …). This can be done by right-clicking the data grid and by choosing the Export to ASCII file (see Figure 4.205).

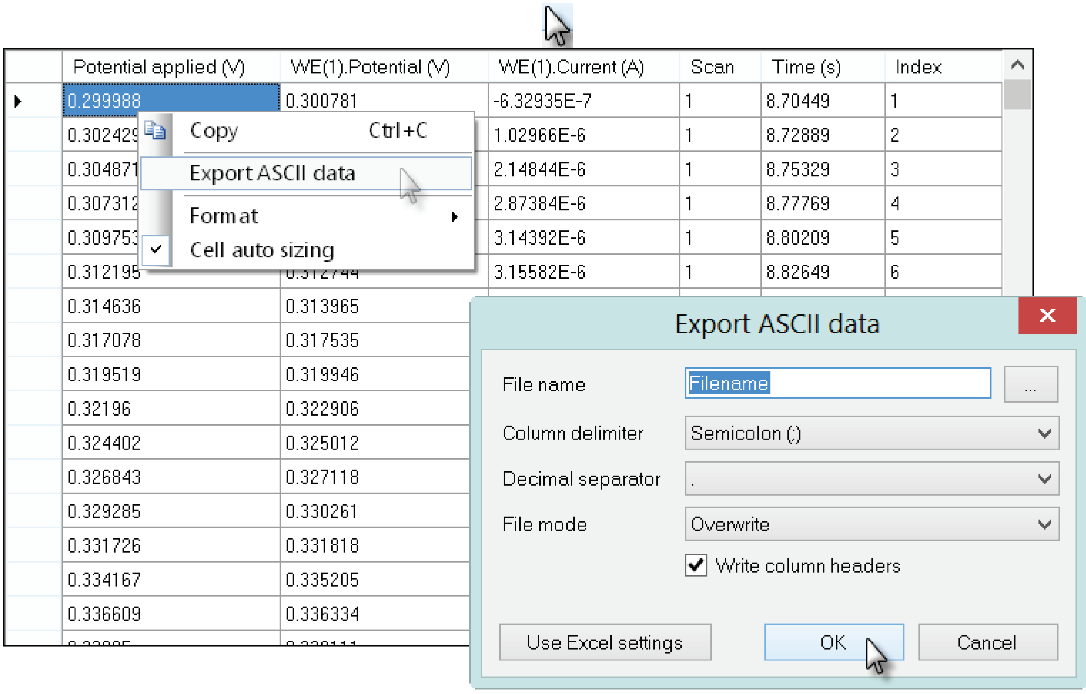


Figure 4.205 – Exporting the data to ASCII

4.10 – The Calculate signal tool

The analysis view comes with a **Calculate signal** tool that can be used to calculate new signals using the values of the available signals. This can be done through the dedicated *Calculate signal* window available in the Analysis view. Clicking the calculator button located in the toolbar on the right-hand side of the data grid opens the Calculate signal interface (see Figure 4.206).

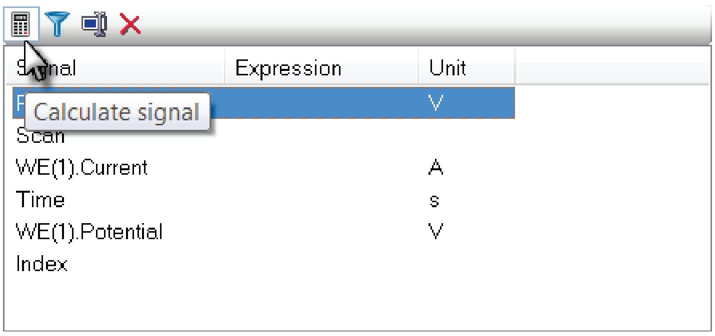


Figure 4.206 – Clicking the button allows to calculate new signals

Note

It is also possible to open the Calculate signal tool by clicking the CV staircase item in the data explorer frame and clicking the button in the quick access toolbar (see Figure 4.207).

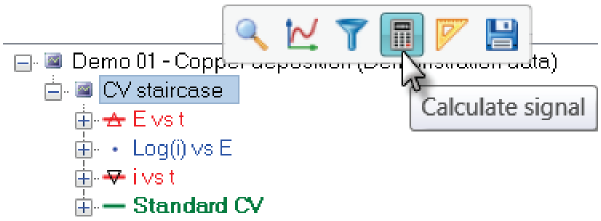


Figure 4.207 – The calculate signal tool is also available from the quick access toolbar

Clicking the button will display the calculate signal window, which can be used to define a new signal and the required expression to calculate it (see Figure 4.208).

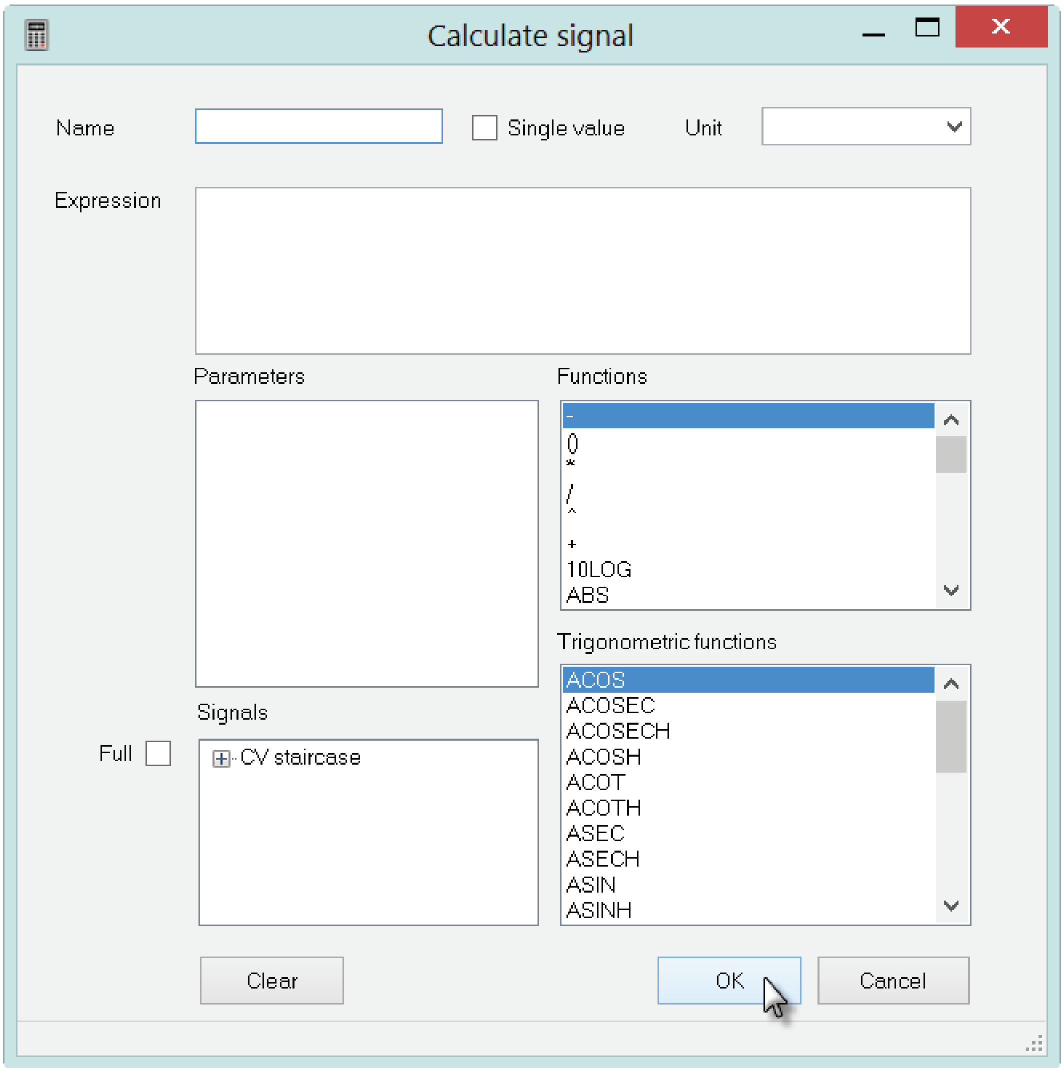


Figure 4.208 – The calculate signal window

The calculate signal window contains several fields.

- **Name:** each signal created using the expression builder must have a unique name.
- **Unit:** the unit of the calculated signal. The unit can be selected from a drop-down list or can be entered manually.
- **Expression:** this field is used to build the new signal using mathematical functions and trigonometric functions, as well as parameters.
- **Parameters:** this field displays the parameters of the expression.
- **Functions:** this field proposes a list of typical mathematical operators that can be used to perform calculations on the data points of a given signal.
- **Trigonometric functions:** a list of trigonometric operators is proposed in this field, to be used in the same way as the mathematical operators.
- **Signals:** this field displays the list of available signals that can be used in the signal creation process. If the data set contains more than one measurement command, it is possible to click the **Full** checkbox in order to display all the available electrochemical signals.

The available signals for the cyclic voltammogram from example #1 are Potential applied, WE(1).Current, Time, WE(1).Potential, scan and Index.

For plotting purposes, it might be useful to express the applied potential relative to the Standard Hydrogen Electrode (SHE). The cyclic voltammogram was recorded using an Ag/AgCl (KCl saturated) reference electrode which has a potential of -0.197 V relative to the SHE at 25°C. In order to change the potential scale, 197 mV has to be added to the Potential applied signal.

In the expression builder window, click the Name field and enter *Potential vs SHE* as a name. Select the V unit from the Unit list. Click the expression field and type the following expression:

$$E + 0.197$$

Where E is the only parameter in the expression. The parameter E corresponds to the Potential applied signal. Click the parameters field to update the list of parameters. Since there is only one parameter, the field will only display E (see Figure 4.209).

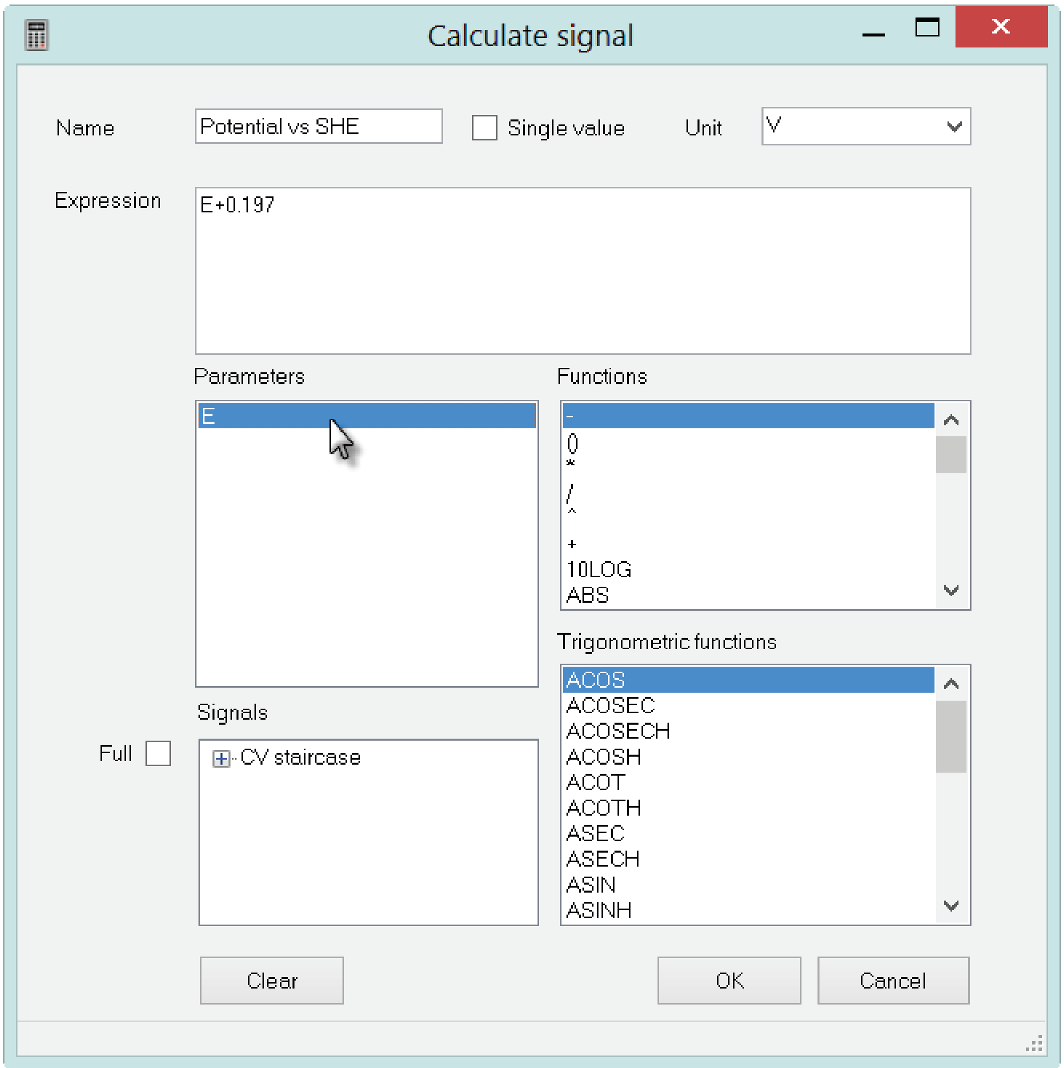


Figure 4.209 – Creating a new signal by adding 197 mV to the applied potential signal

The final step required to calculate a new signal is to select which signals should be used for each parameter. Click the E parameter from the list and click the button next to CV staircase group in the Signals list. This will display all the available electrochemical signals provided by the CV staircase measurement command. Double click the Potential applied signal to assign the values of this signal to the parameters E (see Figure 4.210).

Note

Once the values of a signal have been assigned to a parameter of the expression builder, the name of the parameter will be displayed next to the signal, in between brackets (see Figure 4.210).

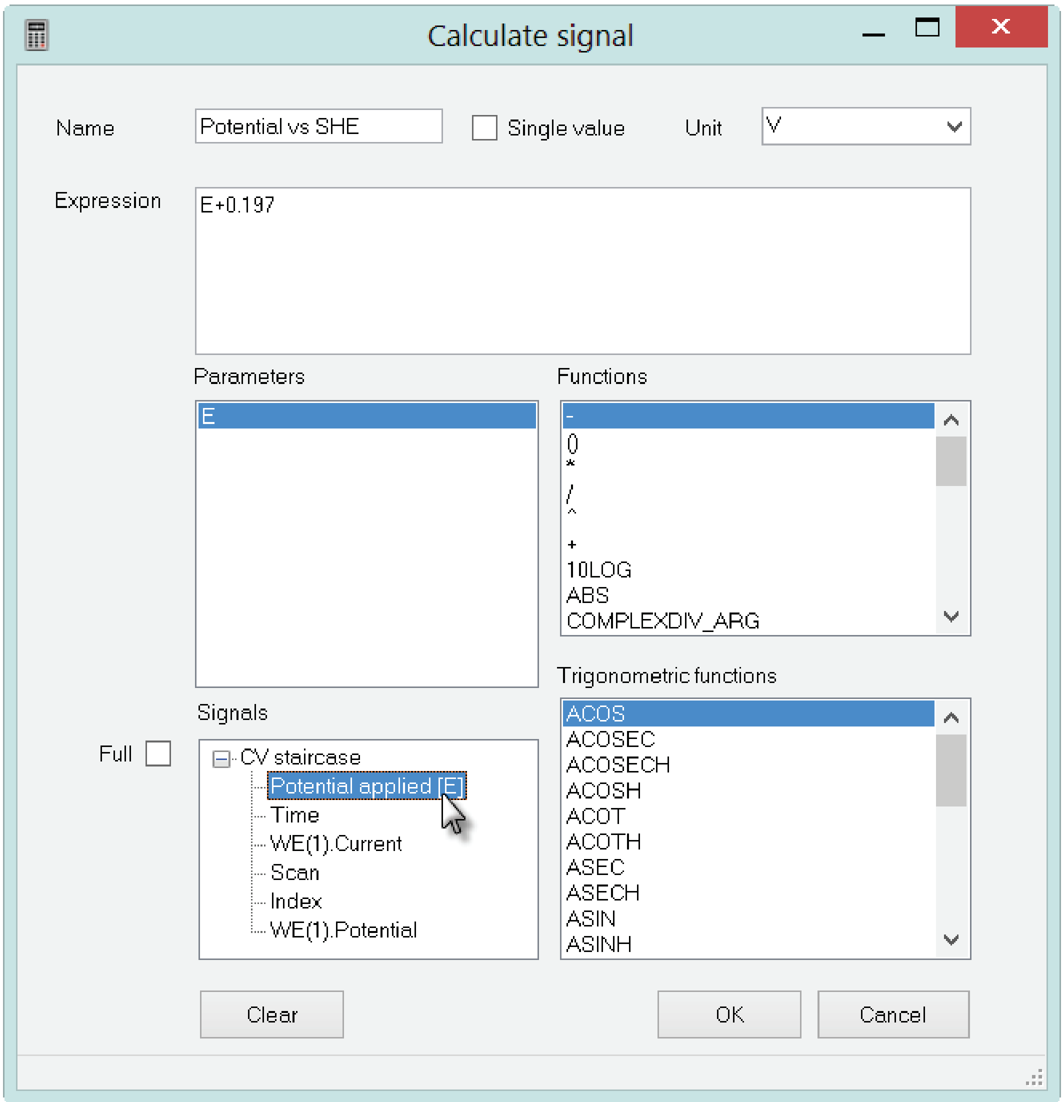


Figure 4.210 – Selecting the values of the Potential applied signal for the parameter E

Click the **OK** button to finish the calculation of the new signal.

After the new signal has been created, its values will be displayed in a new column in the data grid. The corresponding formula will appear under the data grid toolbar on the right-hand side of the screen (see Figure 4.211).

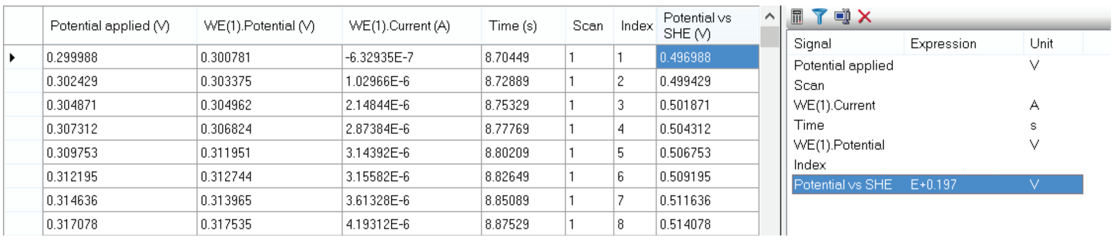


Figure 4.211 – The newly created signal values shown in the data grid. The matching expression is shown on the right-hand side

Having created a new signal for the experimental data from example #1, the Standard CV plot settings can be edited. Right-clicking the settings for the X-axis of the Standard CV plot, change the plot settings from Potential applied to the newly created *Potential vs SHE* (see Figure 4.212).

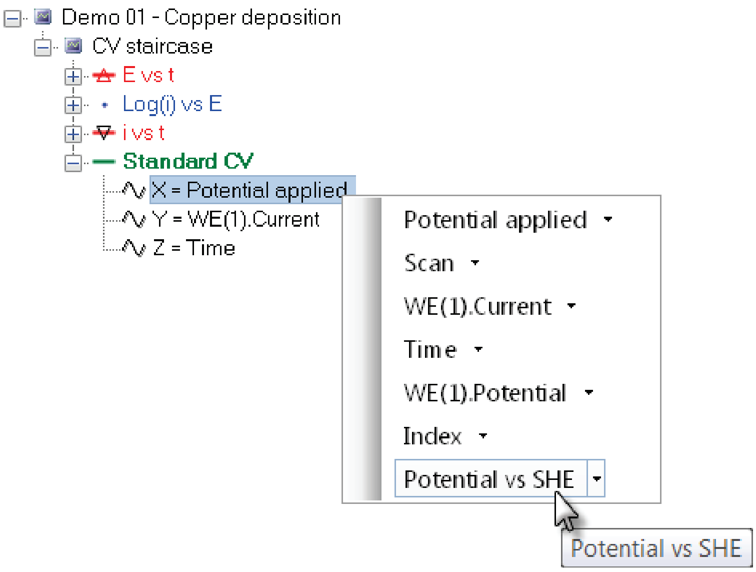


Figure 4.212 – Creating a new plot using the modified potential scale

The new 2D plot should now look like the one displayed in Figure 4.213.

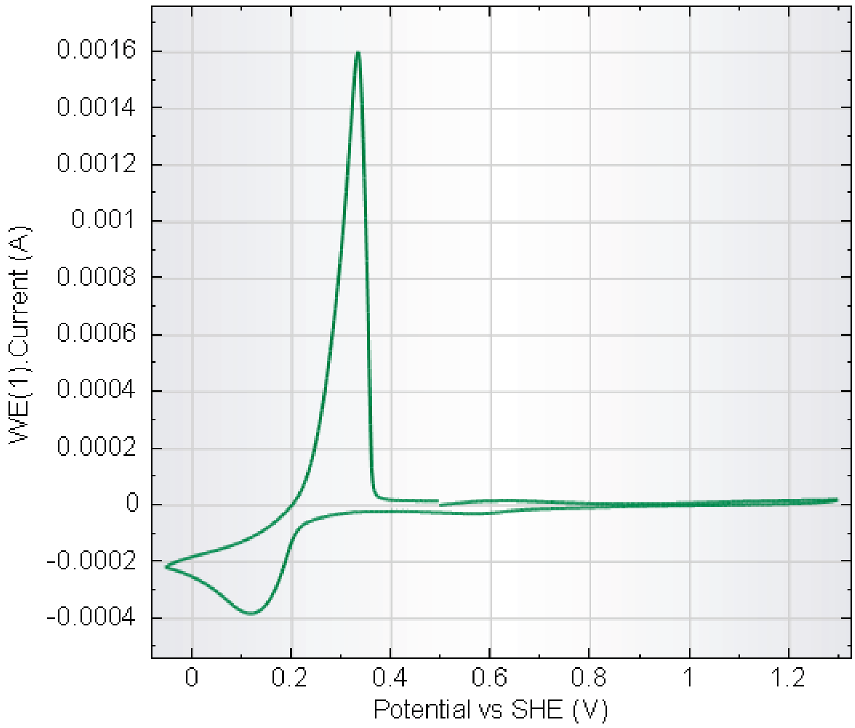


Figure 4.213 – Plotting the data from example #1 using the corrected potential scale

The number of values in the calculated signal is always equal to the number of values in the signal used as expression parameter. If more than one signal is used in the calculation, the signals used in the mathematical expression should all have the same length. If the Single value checkbox is checked in the Calculate signal window, the result of the calculation will be reduced to a single value (the first value of the calculated signal).

Figure 4.214 shows an example of a single value calculation. The calculate signal tool is used to determine the maximum value of the current signal. The Single value checkbox is checked, forcing the calculate signal tool to return a single value.

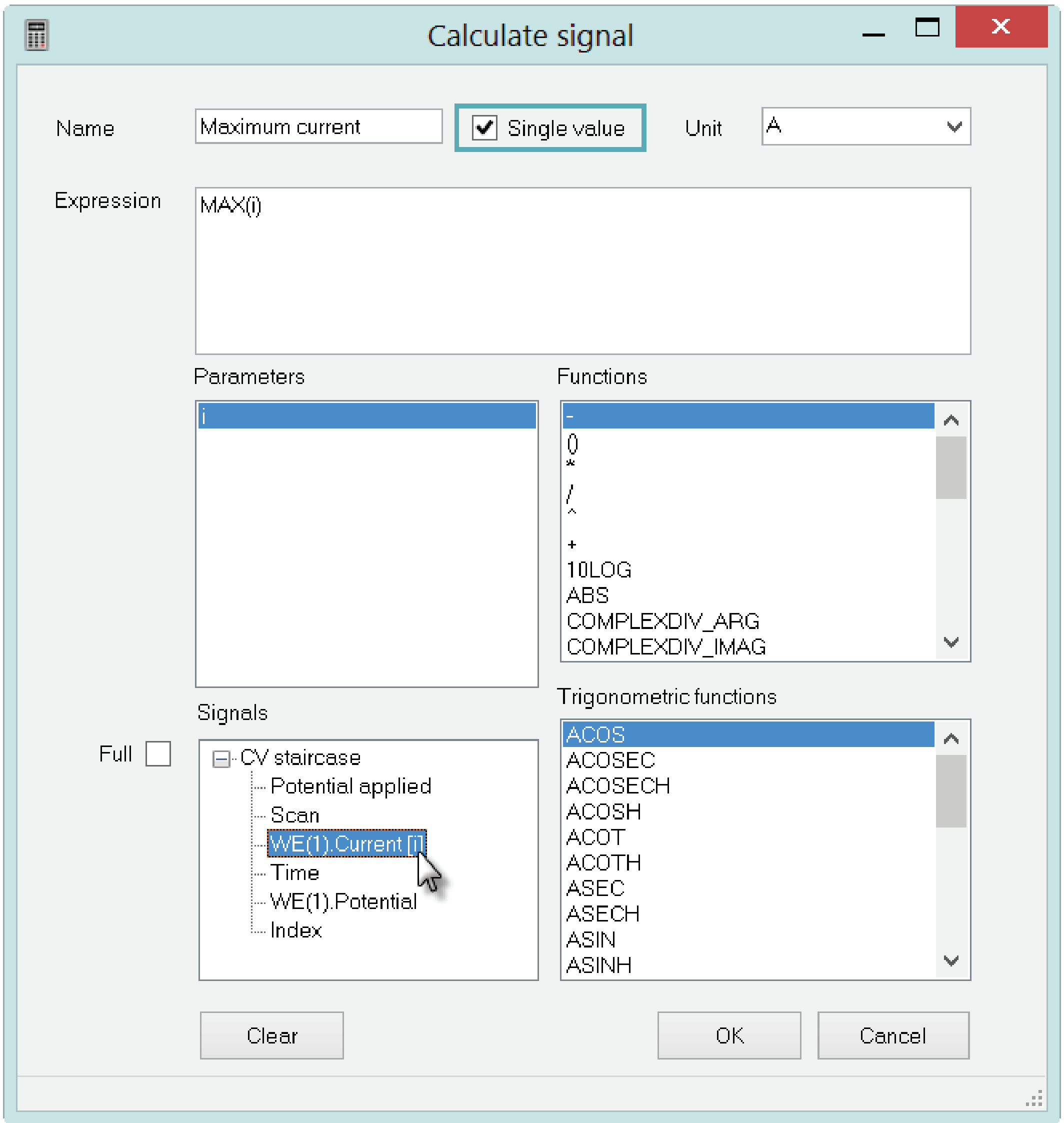


Figure 4.214 – Using the Single value check box reduces the result of the calculate signal to a single value

Note

Single values are very convenient because they can be used as parameters in the procedure editor.

4.11 – The Build signal tool

The calculate signal provided in the analysis view is a convenient tool for create any number of new signals. However, in some cases, it might be necessary to extract measured values from other measurements as well as some of the procedure parameters and use them to create specific plots. This can be done using the **Build signal** tool provided in the analysis view.

Load the Demo 05 data set into the data explorer frame. You will notice that the data set contains four different entries (see Figure 4.215)

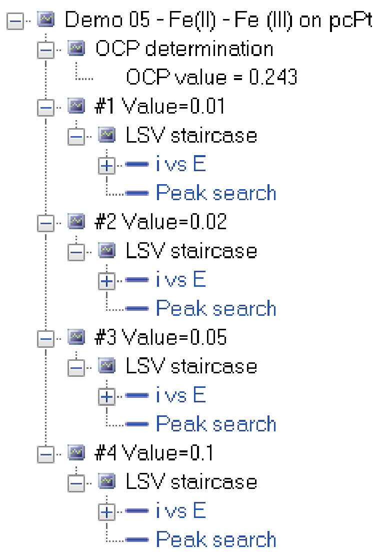


Figure 4.215 – The four LSV curves of the Demo 05 data set

The Demo 05 data set was obtained using the **Repeat for each value** command, with four values used in procedure for the scan rate using in the LSV staircase measurement. The first LSV was obtained with a scan rate of 0.01 V/s, the next LSV was recorded with a scan rate of 0.02 V/s, and so on.

The overlay plot of all four LSV curves is provided below (see Figure 4.216).

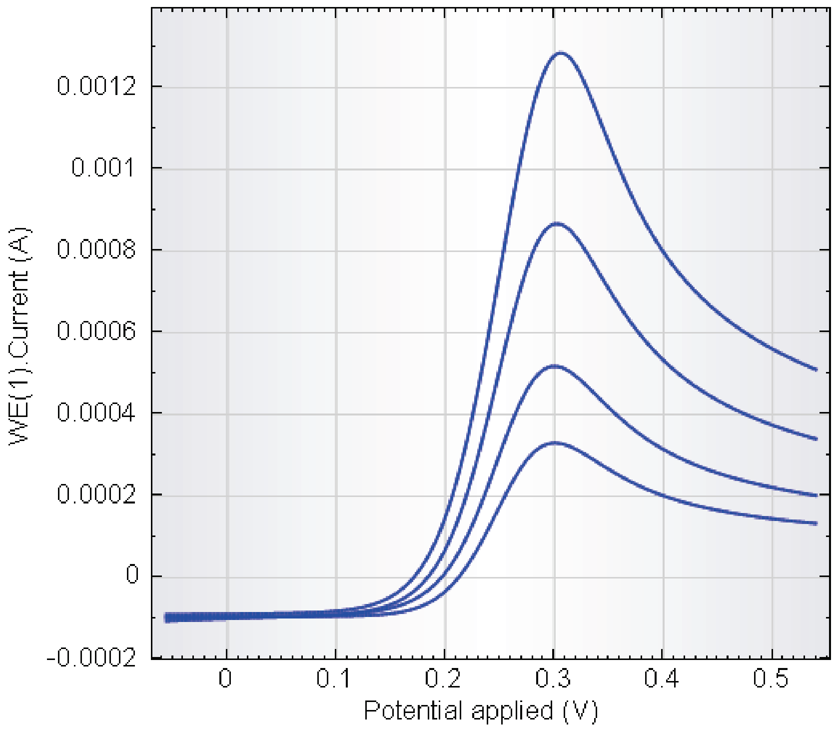


Figure 4.216 – The four LSV curves obtained using four different scan rates

As the scan rate increases, the anodic current increases. For a reversible system, like the $[Fe(CN)_6]^{4-}$ / $[Fe(CN)_6]^{3-}$ system, the peak current, i_p is proportional to the square root of the scan rate:

$$i_p = 2.69 \cdot 10^5 A n^{\frac{3}{2}} C^{\infty} D^{\frac{1}{2}} \nu^{\frac{1}{2}}$$

where A is the area of the electrode, n is the number of electrons, F is Faraday's constant, C^{∞} is the bulk concentration of the electroactive species, D is the diffusion coefficient and ν is the scan rate.

A plot of the peak current vs the square root of the scan rate should yield a straight line. The procedure used to measure the data points of the Demo 05 example included an automatic peak search command. Therefore, the peak height and peak position is available for each individual curve in the data set.

The data grid provides a tool, called the **Build signal**, which is designed to create such a plot, by **filtering** and **selecting** information coming from the available signals and the procedure parameters.

To create the plot, the following information must be extracted from the measured data and the procedure:

1. The scan rate used for each curve
2. The peak height of each peak

Furthermore, it will be necessary to determine the square root of the scan rate.

The use of the signal builder will be illustrated for the construction of the first two items. The third item can be created using the Calculate signal tool described in the previous section.

4.11.1 – The signal builder window

Click the Demo 05 data set in the data explorer to select the whole data set. To create a new signal using procedure parameters or a specific set of values of an electrochemical signal, click the build signal button in the data grid toolbar (Figure 4.217).

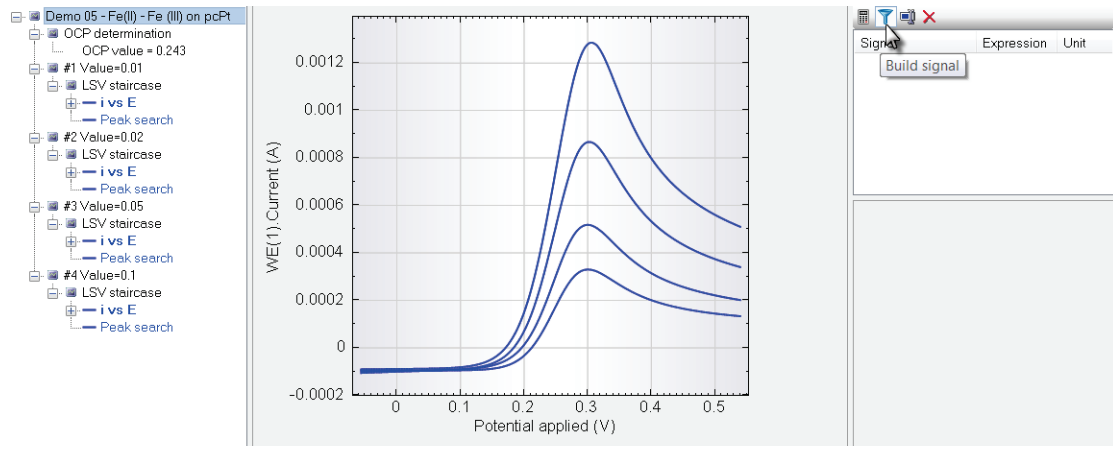


Figure 4.217 – Select the whole data set and click the button in data grid toolbar to use the signal builder

This will open the Filter and Select Signals window (see Figure 4.219).

Note

It is also possible to open the Calculate signal tool by clicking the CV staircase item in the data explorer frame and clicking the button in the quick access toolbar (see Figure 4.218).

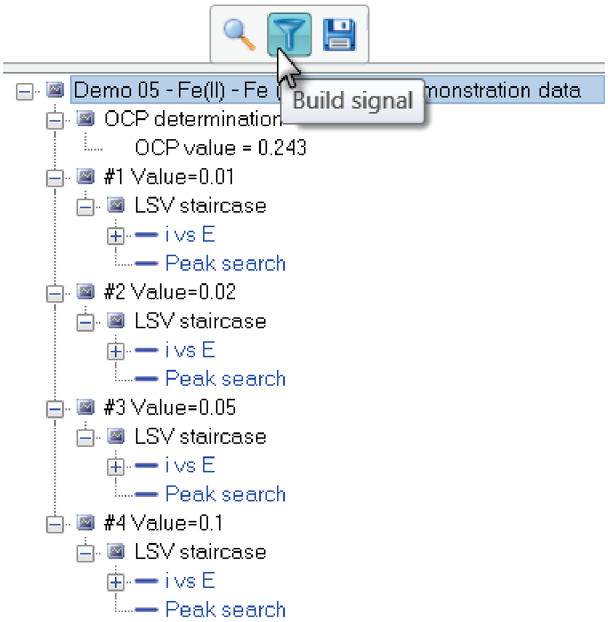


Figure 4.218 – The build signal tool is also available from the quick access toolbar

Note

Since the new signals use values of all the measurements in the data set, clicking the header is required.

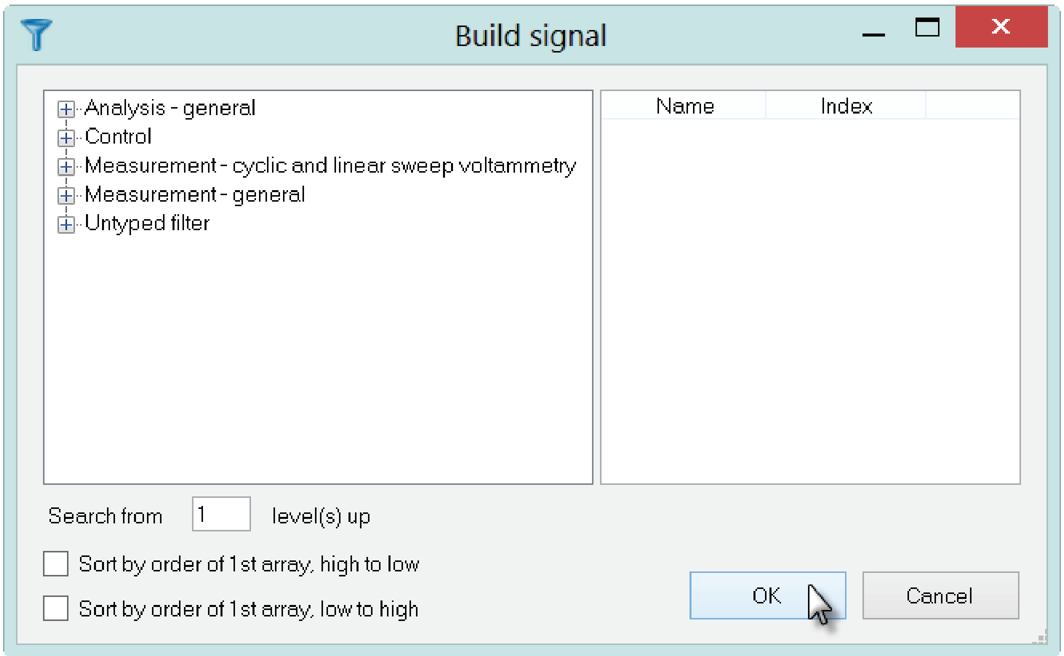


Figure 4.219 – The filter and select signals window

The Filter and select signals window has two frames. The frame on the left contains five groups. Each group contains the individual commands used in the procedure, along with the electrochemical signals and the parameters. The frame on the right is empty. It has two columns, Name and Index. A sorting option and a search depth are available at the bottom of the window.

The signal builder can be used to build new signals by **filtering** and **selecting** measured data points or procedure parameters, using user-defined criteria.

4.11.2 – Selecting the values of the scan rate

The scan rate values used in the experiment were defined using the *Repeat for each value* command, linking the values of this command to the scan rate value of the *LSV staircase* command (see Figure 4.220).

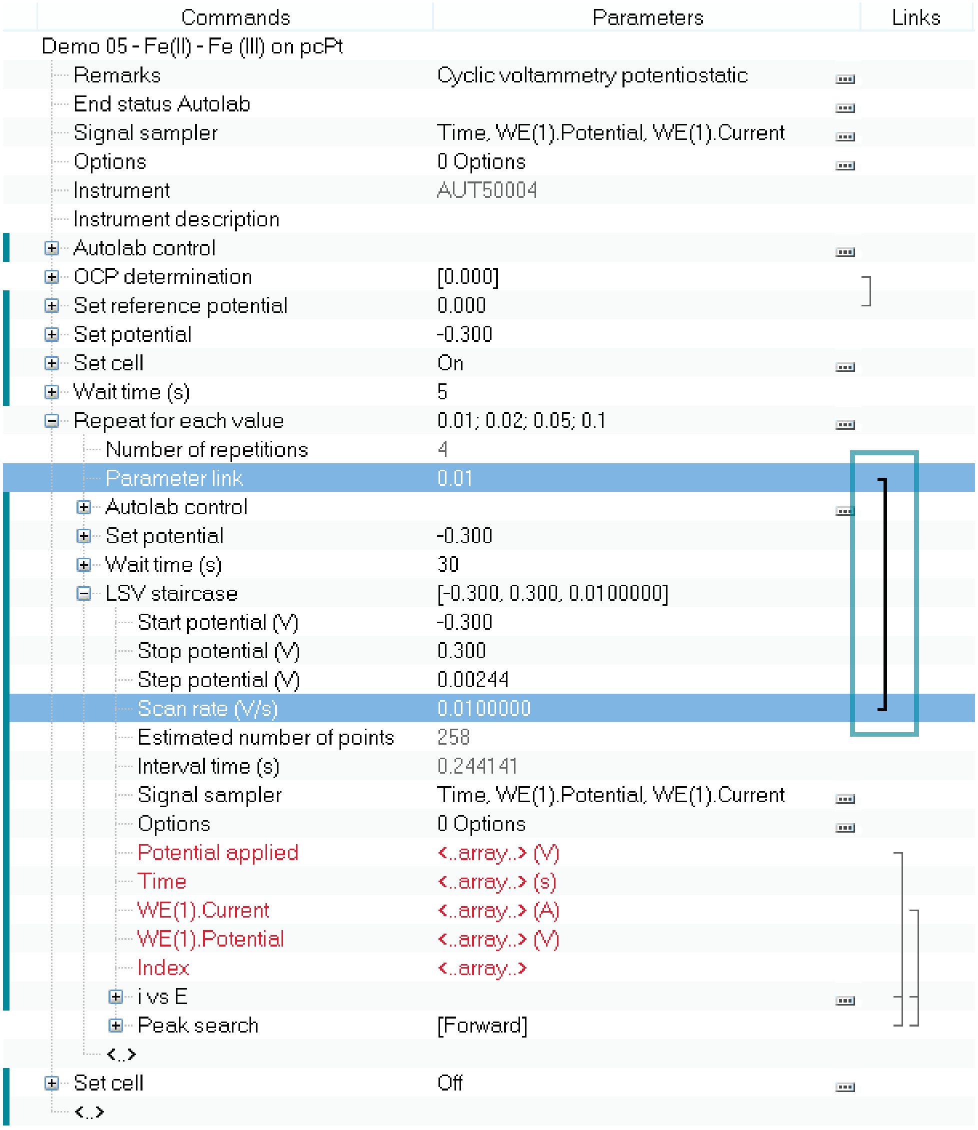


Figure 4.220 – Detailed view of the Repeat for each value linked to the Control external device command

Expand the *Measurement – cyclic and linear sweep voltammetry* group in the left frame, and then expand the LSV staircase item. Eight items will be displayed (see Figure 4.221):

- Index
- Potential applied
- Scan rate (V/s)
- Start potential (V)
- Stop potential (V)
- Time
- WE(1).Current
- WE(1).Potential

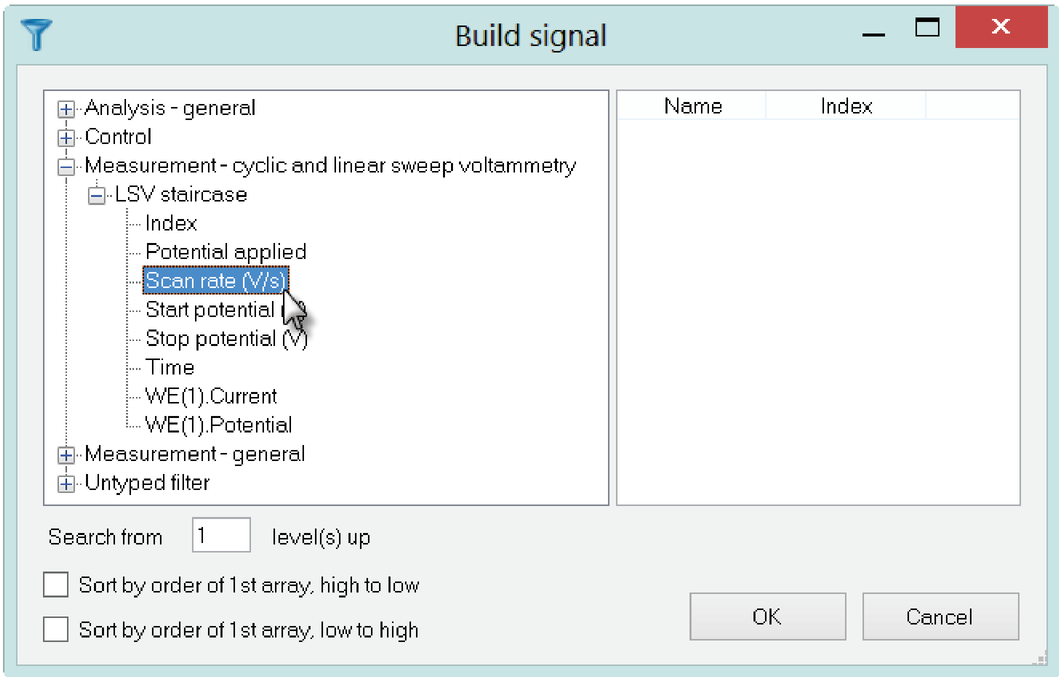


Figure 4.221 – Adding the value of the scan rate to the filter

Double click the Scan rate (V/s) located under the LSV staircase group to add it to the list in the frame on the right-hand side (Figure 4.222).

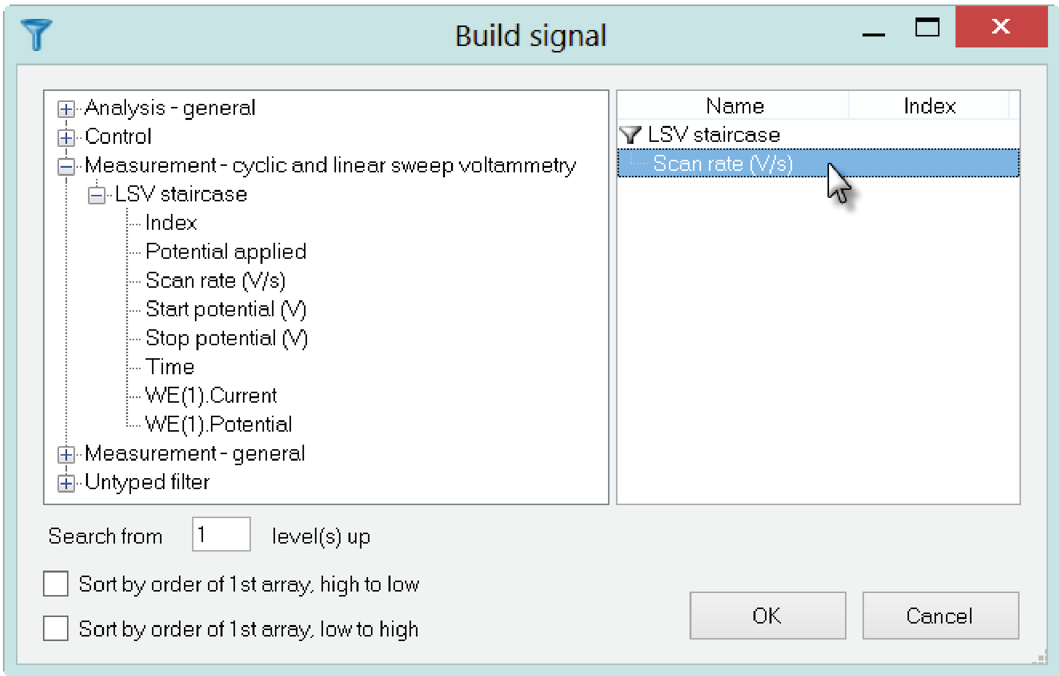


Figure 4.222 – Double click the scan rate item to add it to the filter and select list

Clicking the OK button generates a new signal, called **Scan rate (V/s)** in the data grid. This signal contains four values (see Figure 4.223).

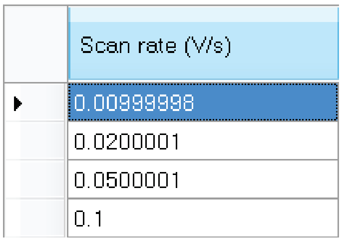


Figure 4.223 – The Scan rate signal

The signal obtained using the signal builder contains all the scan rate values used in the LSV staircase command.

4.11.3 – Selecting the values of the peak height

The second signal which is required for the plot is a signal containing the values of the peak height. For this example, a filter designed to extract the values of the peak height for curve will be constructed.

Click the Build signal button to open the Filter and select signals window. In the Analysis – general, locate the Peak height in the Peak search list. Double click the Peak height item to add it to the filter (see Figure 4.224).

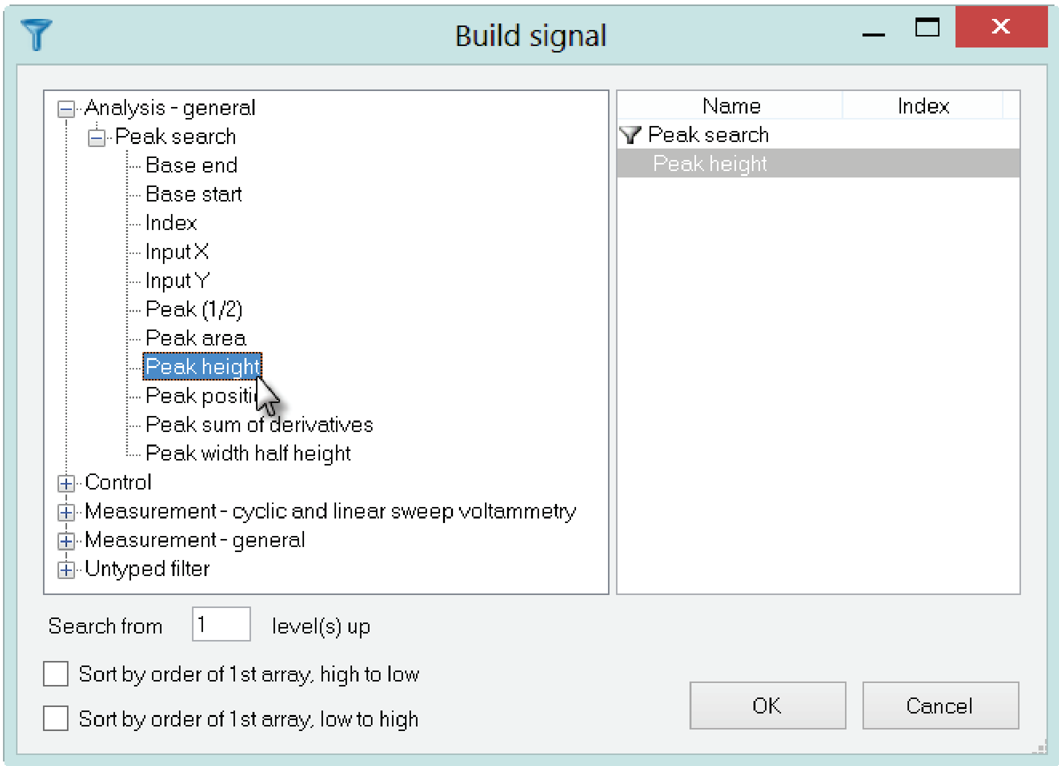
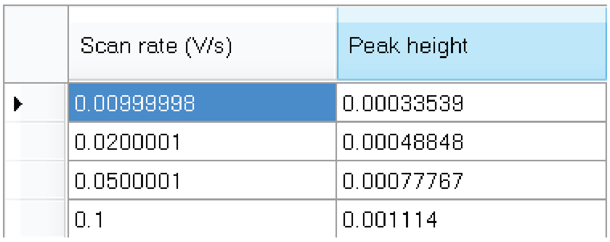


Figure 4.224 – Adding the Peak height from the Peak search group to the filter

Click the OK button to validate the filter. A new signal will be created in the data grid, containing the values of the peak height for each LSV Staircase curve of the data set (see Figure 4.225).



Scan rate (V/s)	Peak height
0.00999998	0.00033539
0.0200001	0.00048848
0.0500001	0.00077767
0.1	0.001114

Figure 4.225 – The data grid and the two filtered signals

4.11.4 – Calculating the square root of the scan rate

For the construction of the plot, a final signal is required, containing the square root of the scan rate.

In order to create this signal, click the button located in the toolbar to use the Calculate signal tool. In the Calculate signal window, create a *Square root scan rate* signal using the following expression:

SQRT(v)

It is possible to copy and paste the expression displayed above into the expression builder.

In the signal list, define the scan rate signal on top of the list as the *v* parameter by double clicking the scan rate signal.

Click the OK button to create the new signal (see Figure 4.226).

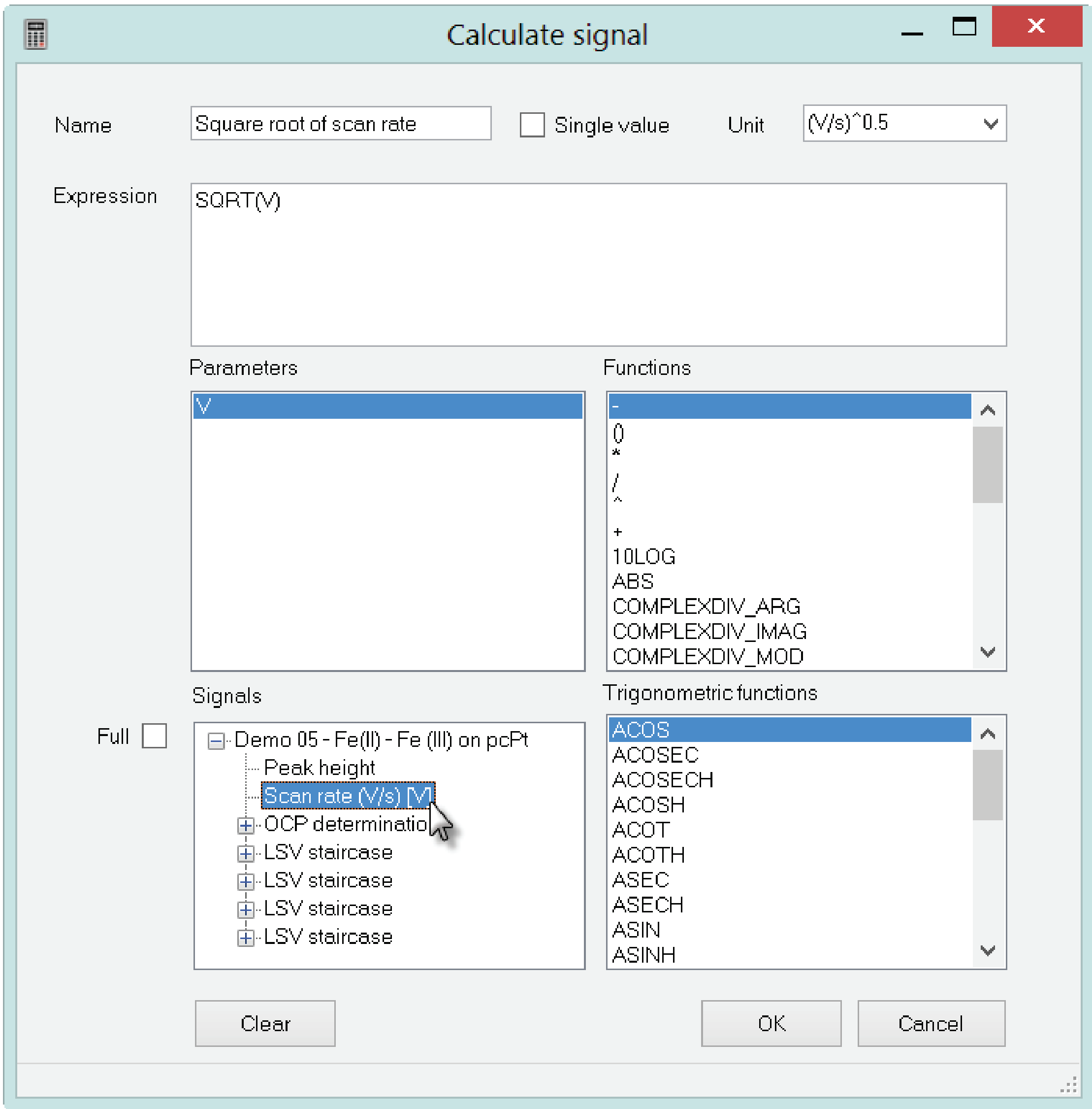


Figure 4.226 – Creating the square root of the angular frequency signal

Three signals should now be available in the data grid (see Figure 4.227).

	Scan rate (V/s)	Peak height	Square root scan rate ((V/s)^1/2)
▶	0.00999998	0.00033539	0.0999999
	0.0200001	0.00048848	0.141422
	0.0500001	0.00077767	0.223607
	0.1	0.001114	0.316228

Figure 4.227 – The signals required to build the plot

4.11.5 – Building the plot

With all the required signals available, a plot to test the reversibility of the electron transfer can be added to the data set. First click the 2D plot button and then right-click the header of the data set in the data explorer frame and select the Add Plot – Custom option from the menu (see Figure 4.228).

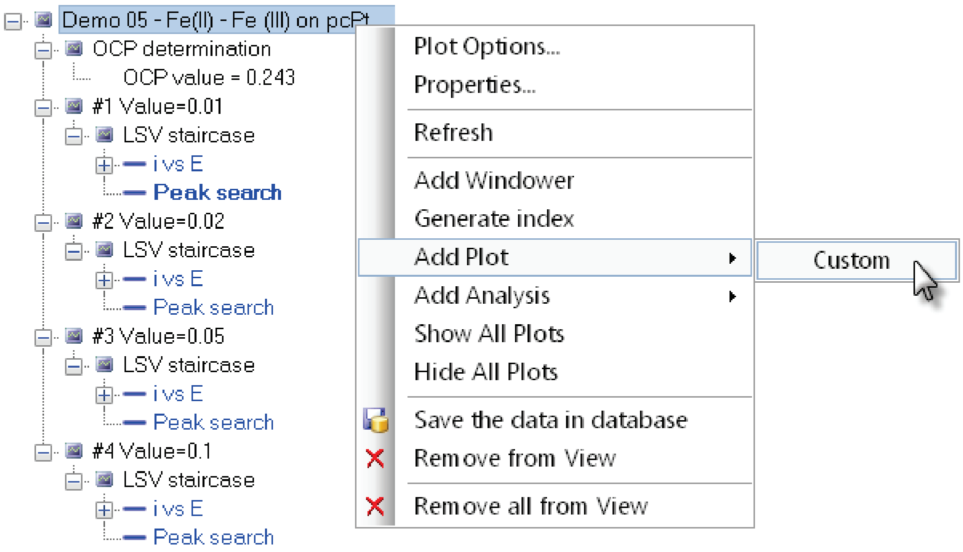


Figure 4.228 – Adding a Custom plot to the data set

A new custom plot will be added to the data explorer frame. Set the X-axis signal to Square root scan rate and the Y-axis signal to Peak height. Rename the plot to Reversibility plot.

Note

Only three signals are defined for the whole data set (Scan rate, Square root scan rate and peak height). Therefore, only these three signals can be used for the plot (see Figure 4.229).

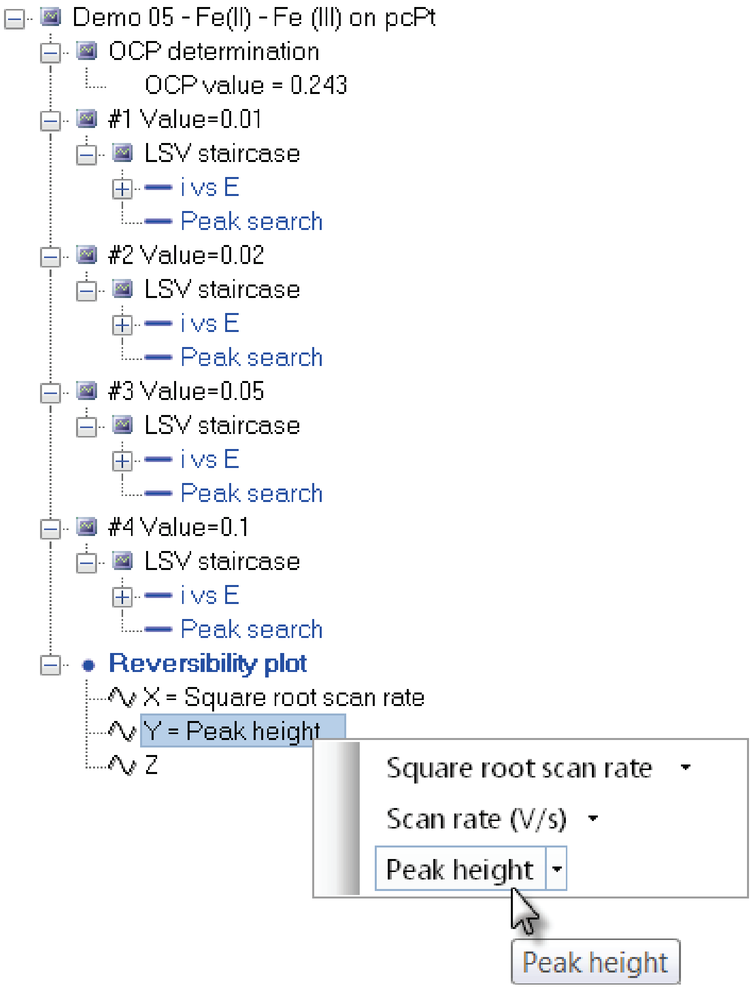


Figure 4.229 – Defining the plot settings for the custom plot

Figure 4.230 shows the resulting plot, after changing the plot settings to a Point plot, using a size 8 diamond as a marker. As expected, the points align on a straight line.

Note

The show origin option is used in the plot.

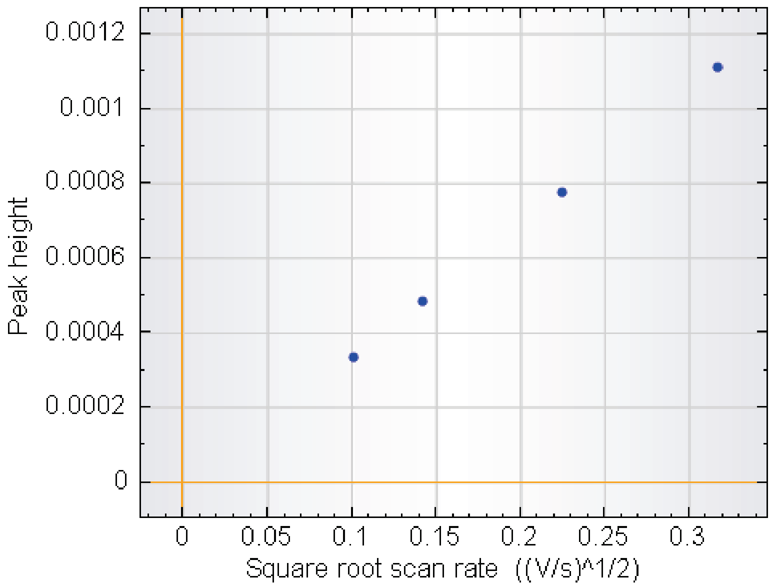


Figure 4.230 – The completed reversibility plot

4.12 – The windower

The windower is an additional tool that can be used for data analysis. The role of the windower is to extract a cross section of the data grid. It is designed to work in the same way as the signal builder: it creates a new set of data based on measured or calculated signals.

The windower can be used to select a scan from a series of cyclic voltammograms, or select a potential step from a chrono amperometry experiment or select only the data points with positive current values.

This section will describe the use of the windower using an example from the demo database.

Note

The *Demo 06 – Galvanostatic CV* entry of the demo database contains three galvanostatic staircase cyclic voltammograms for lead deposition recorded using a polycrystalline gold electrode, in 0.1 M $HClO_4$ solution, $[Pb^{2+}]$ = 0.01 M. The reference electrode was a Ag/AgCl (KCl saturated) and the counter electrode was a platinum sheet.

The galvanostatic CV from Demo 06 is displayed in Figure 4.231.

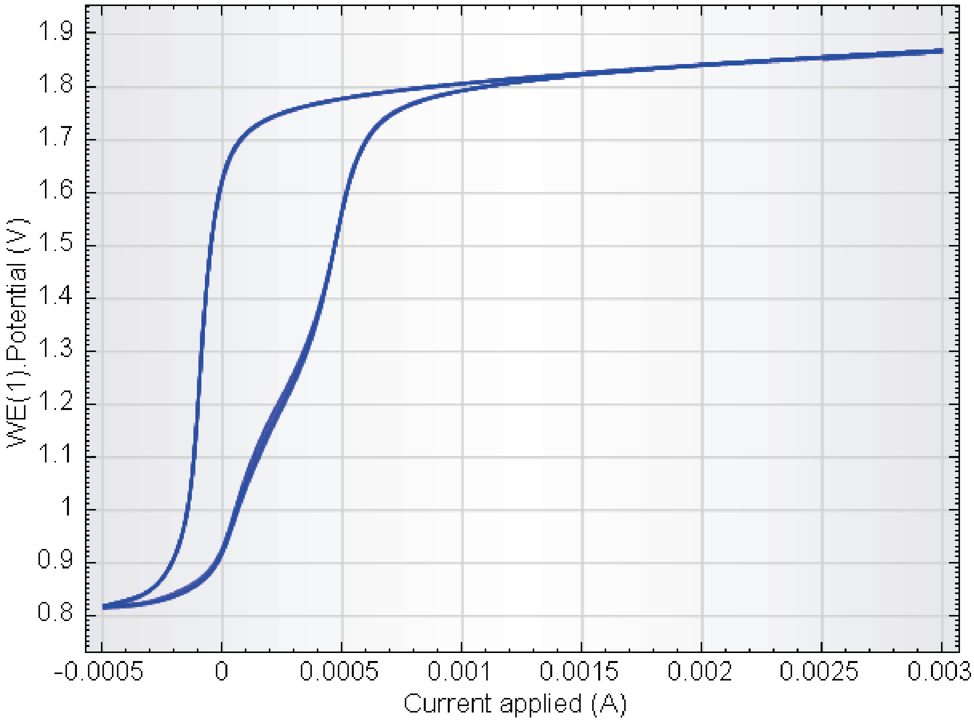


Figure 4.231 – Galvanostatic CV measurement

A total of three scans were recorded in this experiment. The windower will be used in this section to extract the data points from the second scan.

4.12.1 – Adding a windower

To use the windower, which will be used to extract a cross section from the data grid, right-click the CV staircase galvanostatic and select the Add Windower option from the context menu (see Figure 4.232). This will add a new item, called Windower to the data set.

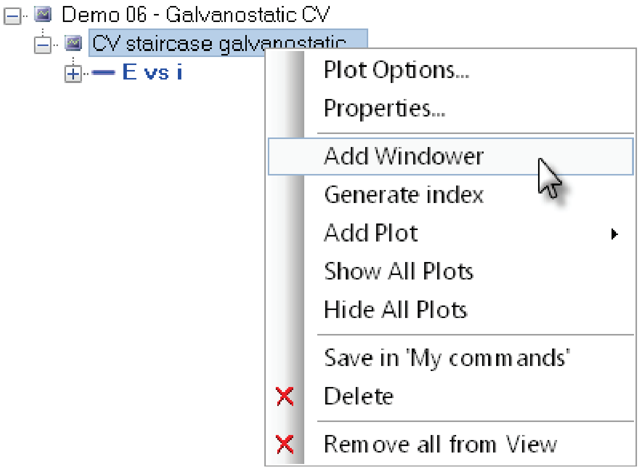


Figure 4.232 – Adding a windower to the CV staircase galvanostatic

It is also possible to open the Windower tool by clicking the CV staircase item in the data explorer frame and clicking the button in the quick access toolbar (see Figure 4.233).

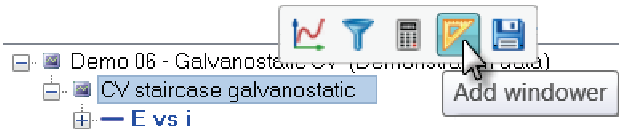


Figure 4.233 – The windower tool is also available from the quick access toolbar

Adding the windower tool triggers the Windower editor window to appear (see Figure 4.234).

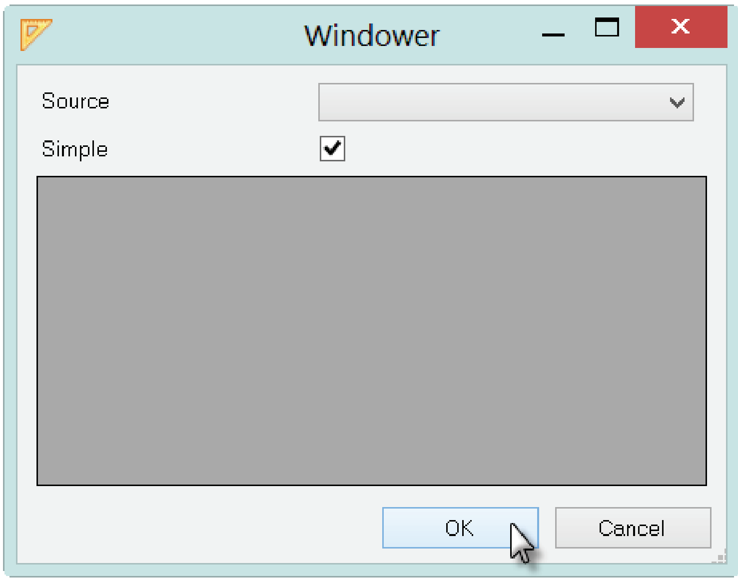


Figure 4.234 – The Windower editor window

The following items need to be specified for the Windower tool to work properly:

- **Source:** this is the source signal used to window the data. The source signal is one of the available signals available in the data set (Time, Scan number, WE(1).Potential, …). Only one source signal can be selected.
- **Simple (checkbox):** this specifies if the simple editor is used (selected by default).
- **Values:** this defines the values of the selected source signal to use in the windower.

4.12.2 – Selecting the source of the windower

The source is one of the available electrochemical signals and will be used to create the cross-section of the data set.

The data set from Demo 06 has the following signals:

- Current applied (A)
- Index
- Scan
- Time (s)
- WE(1).Potential (V)

To select the source for the windower, select the Scan signal from the drop-down list in the editor (see Figure 4.235).

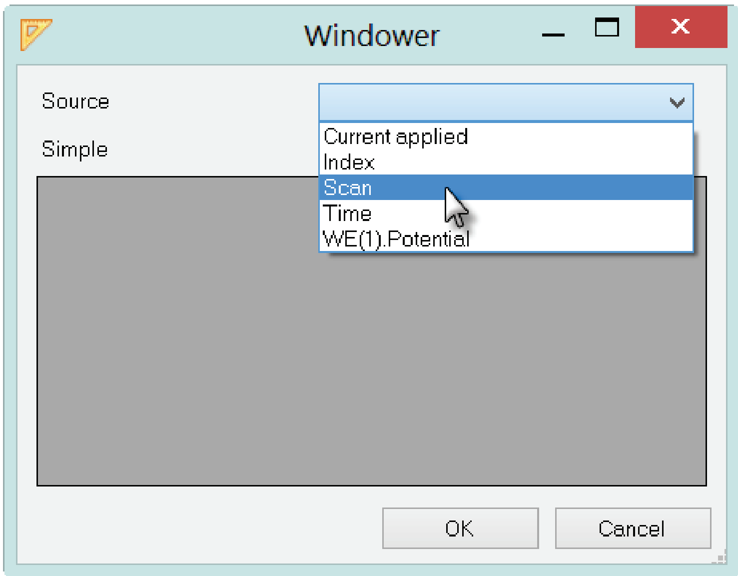


Figure 4.235 – Selecting the Scan signal as the windower source

As soon as the source is defined, the Windower editor displays a list of available values for the selected signal in the frame in the middle (see Figure 4.236). Since this data set only contains three scans, three checkboxes are provided, one for each scan.

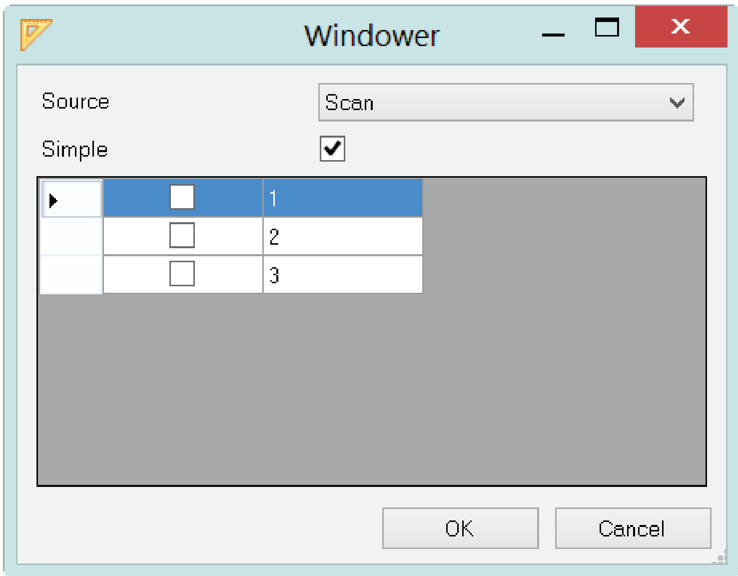


Figure 4.236 – The contents of the Windower editor are updated as soon as the source signal is defined

Select the checkbox corresponding to the second scan in the editor to select the second scan for the windower (see Figure 4.237).

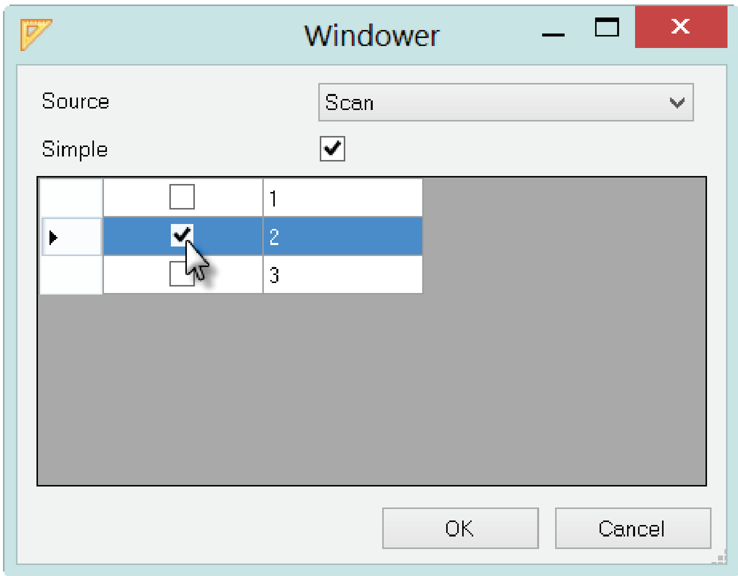


Figure 4.237 – Selecting scan number two in the editor

Click the  button to close the editor and add the specified windower to the data set (see Figure 4.238).

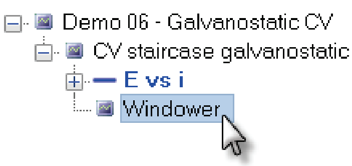


Figure 4.238 – The windower is added to the data set

4.12.3 – Plotting the selected data

Now that the windower has been completely defined, a plot can be added to it. The plot attached to the windower will display only the values in its data grid, in the present example, the recorded data points from scan number two.

To add a plot to the windower, right-click the signal windower in the data explorer and select the **add plot – E vs i** option (see Figure 4.239).

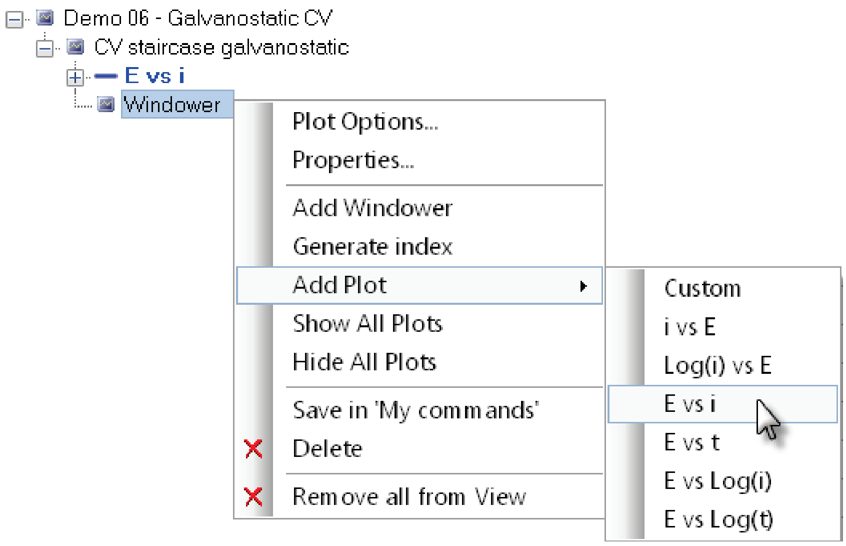


Figure 4.239 – Adding a E vs i plot to the signal windower

Switch to a 2D plot and select the E vs i plot added to the Windower and change the plot options to a size 3 red line plot. The data points from scan number two will be displayed (see Figure 4.240).

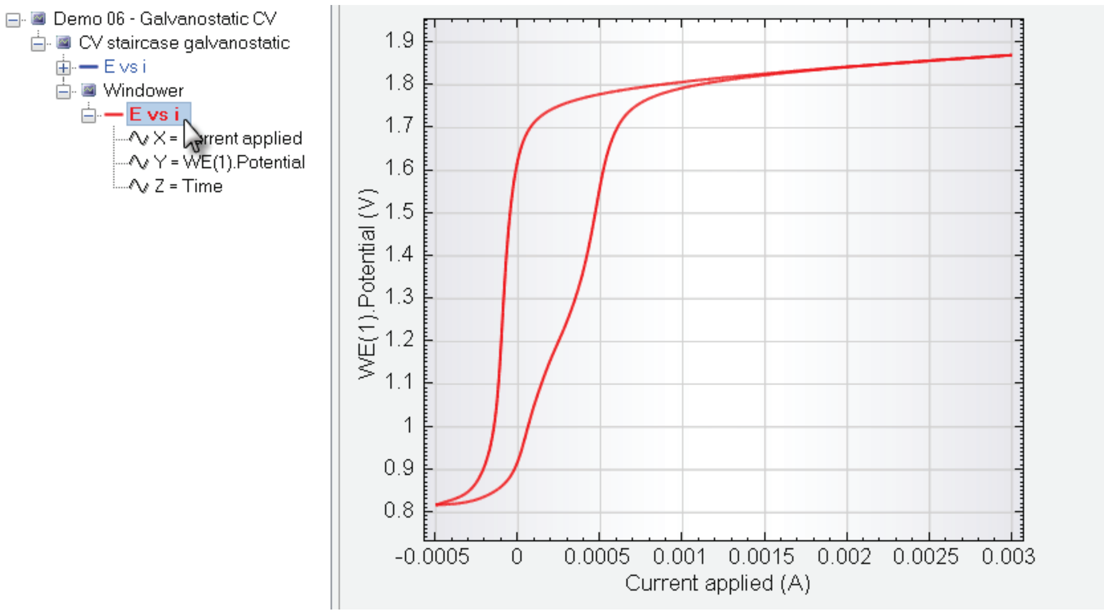


Figure 4.240 – Plotting the data points from scan two

Overlay both E vs i plots using the CTRL-select method and set the time signal on the X-axis for both plots. The resulting overlay plot should look like the one displayed in Figure 4.241 (after changing the X axis to Time).

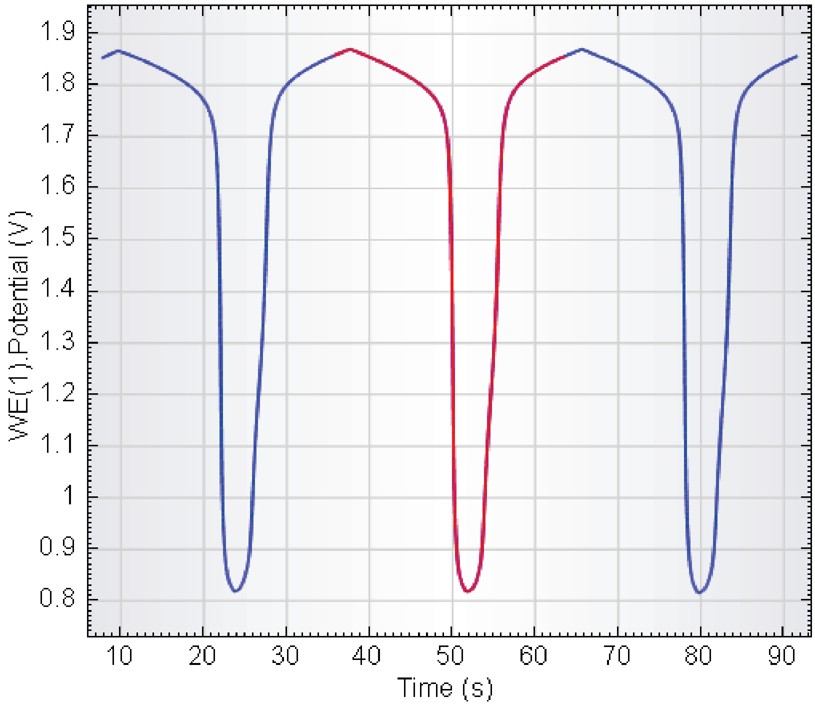


Figure 4.241 – The original data set (WE(1).Potential vs time – blue curve) and scan number 2, selected using the windower (red curve)

Click the added windower item in the data explorer to reveal the control parameters, defined in the editor, on the right-hand side of the 2D plot (see Figure 4.242).

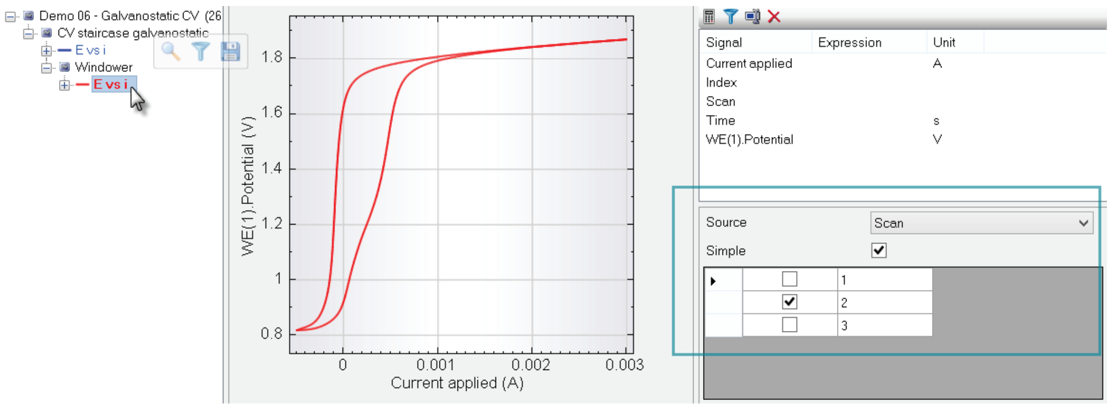


Figure 4.242 – The control parameters of the windower are displayed on the right-hand side of the plot

4.12.4 – Changing the selected data

With the windower selected, it is possible to select more than one value from the list in the control interface. For example, select the third scan by click the corresponding check box. The plot will be immediately updated, displaying the second and third scan in red (see Figure 4.243).

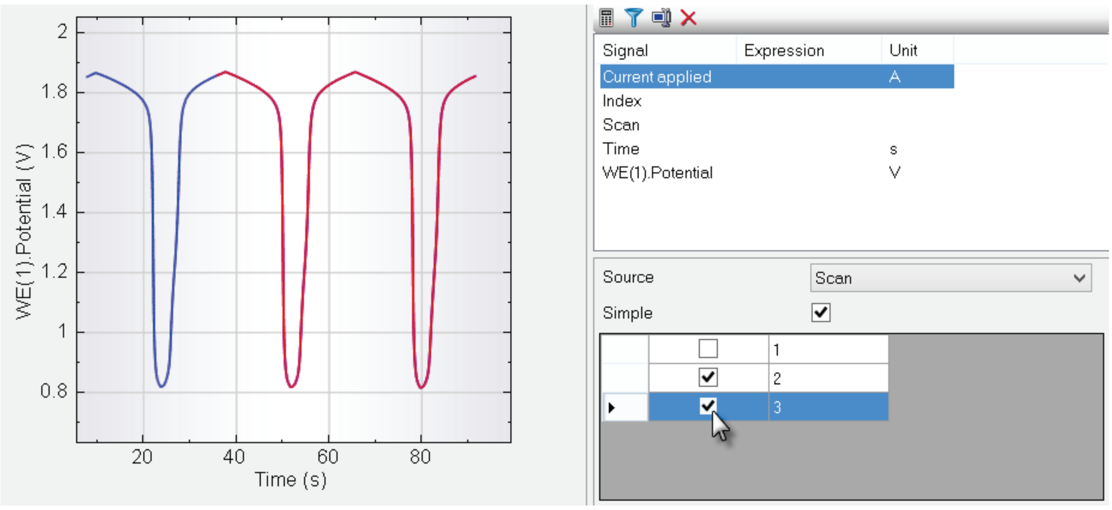


Figure 4.243 – Adding items to the windower will automatically update the selected data

> **Note**
>
> It is possible to use the right-click menu on the list displayed in the control interface to quickly select or deselect all the available check boxes. It is also possible to easily invert the selection (see Figure 4.244).

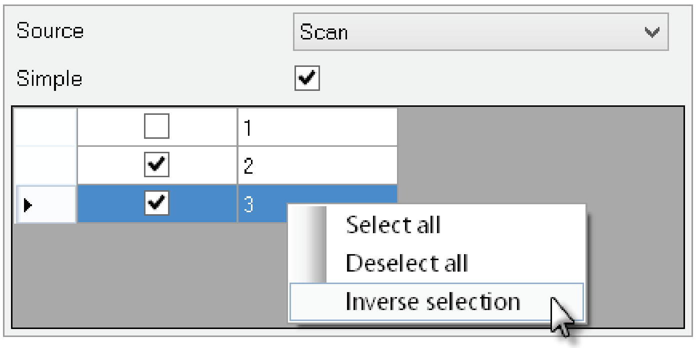


Figure 4.244 – The right-click menu allows you to quickly select or deselect the available check boxes

4.12.5 – Advanced selection

When the *Simple* checkbox is selected, the Windower will always list the possible values for the selected source signal as a list of checkboxes. It is possible to switch to an advanced selection mode by unchecking the Simple checkbox. The selection of the windowed points can then be performed by specifying a value range: begin to end (see Figure 4.245).

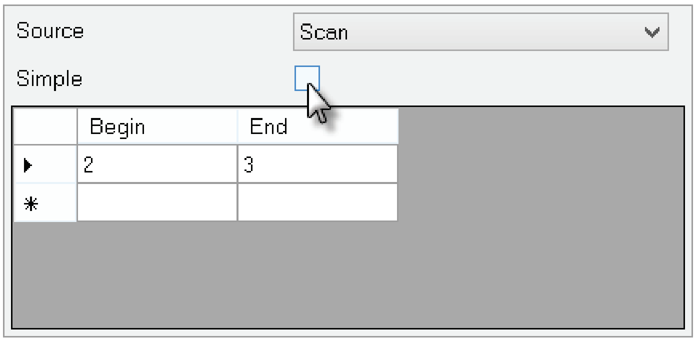


Figure 4.245 – Switching the windower to advanced mode

In Figure 4.245, the selection begins at 2 and ends at 3, which is the same selection as specified in Figure 4.243. This selection mode is useful when the number of available values for the selected signal is very high.

Note

When the number of values for the selected source signal exceeds 300, Nova will automatically switch from *simple* to *advanced*.

Switch the source signal from Scan to Time, using the dropdown list (see Figure 4.246).

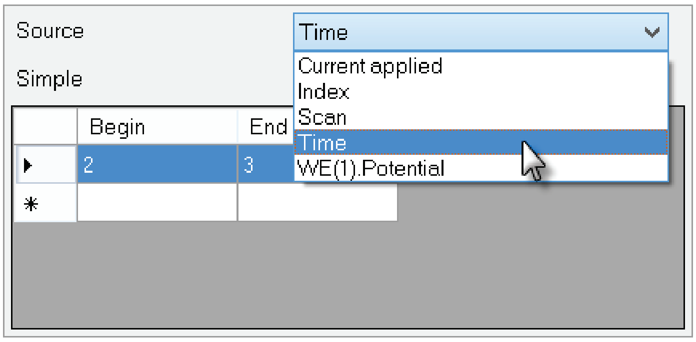


Figure 4.246 – Setting the source signal to Time

Using the advanced selection mode and the Time signal as a source, it is possible to select, for example, all the points measured from 20 s to 40 s (see Figure 4.247).

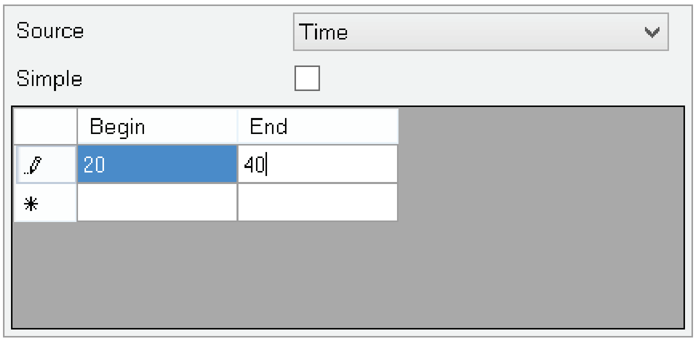


Figure 4.247 – Specifying a range of values

Furthermore, in Advanced selection mode, it is possible to specify several value ranges. Figure 4.248 shows an example in which a second data range has been added to the Windower, from 60 to 80 seconds.

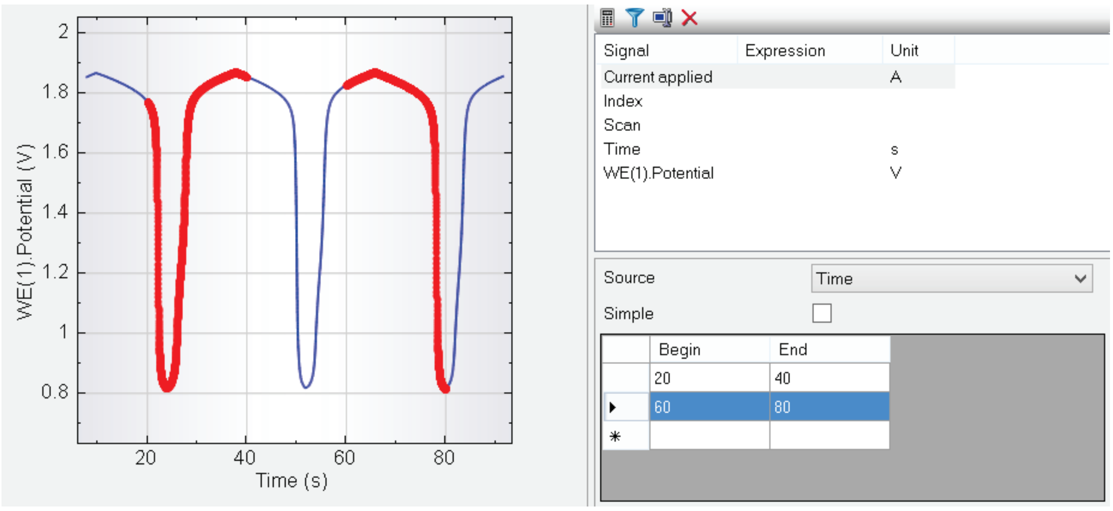


Figure 4.248 – Specifying several ranges in the windower

4.12.6 – Direct windowing through X-axis scaling

It is also possible to window the data immediately, using the X-axis scaling as a selection criterion. This alternative uses the data plotted on the plot area as the data to be windowed. Starting from the E vs t plot obtained from the data provided in the Demo 06 set, one of the different zooming or scale adjustment methods described in this chapter[36] can be used to focus on a particular area of the plot (see Figure 4.249).

[36] Please refer to Section 4.7.9 for more information.

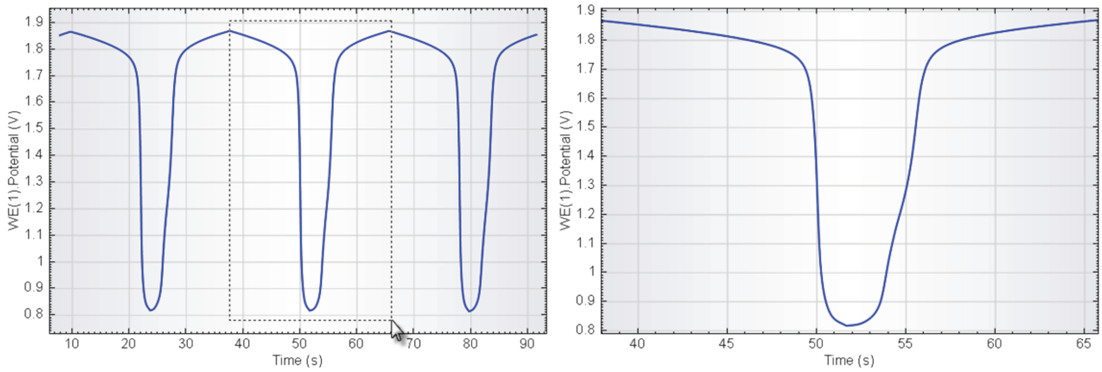


Figure 4.249 – Adjusting the X axis scale in order to graphically select the second scan

With the X axis adjusted, right-click the E vs t plot and select the **Create Windower from X axis** option from the context menu (see Figure 4.250).

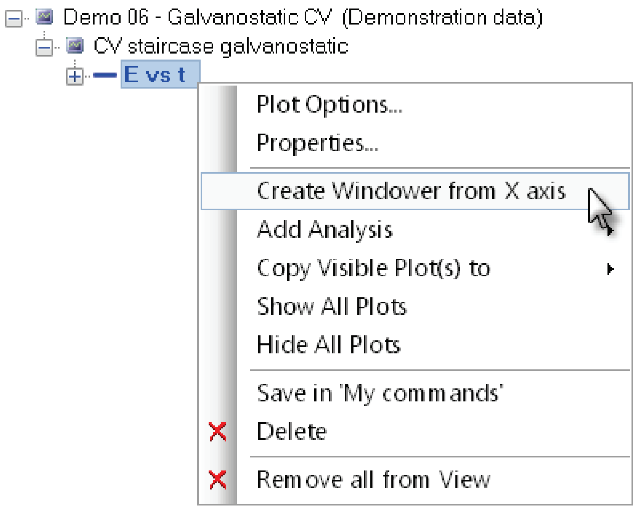


Figure 4.250 – Select the Create Windower from X axis to create a windowed set of data

With the X axis adjusted, right-click the E vs t plot and select the **Create Windower from X axis** option from the context menu (see Figure 4.250). A windower will be added to the dataset (see Figure 4.251). This windower automatically uses the signal plotted on the X axis as the *Source* and the minimum and maximum of the X axis scale as the beginning and end of the windower, respectively.

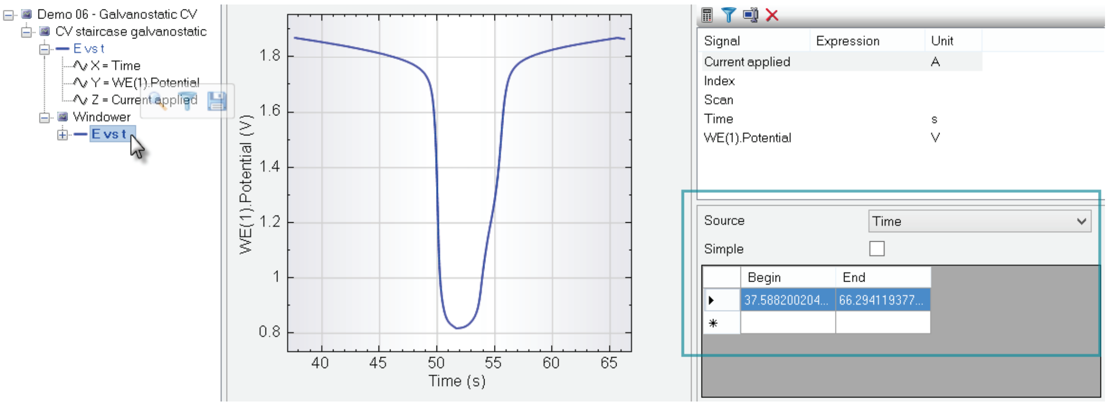


Figure 4.251 – A new windower is added to the data

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