# 14. Chronoamperometry (Supplement)
## 1. Description of the measurements
**(Autolab potentiostat and NOVA controller program)**

1. Turn on the computer and the potentiostat.

2. Connect the appropriate wires to the electrochemical cell. R (blue) - reference electrode (NCE), C (black) counter (auxiliary) electrode (Pt), W (1,2) (red, plugged together) working electrode (Au or GC). There is a green ground, you do not have to use it, but do not touch any electric conductor with it.

3. Start the NOVA controller program on your computer.
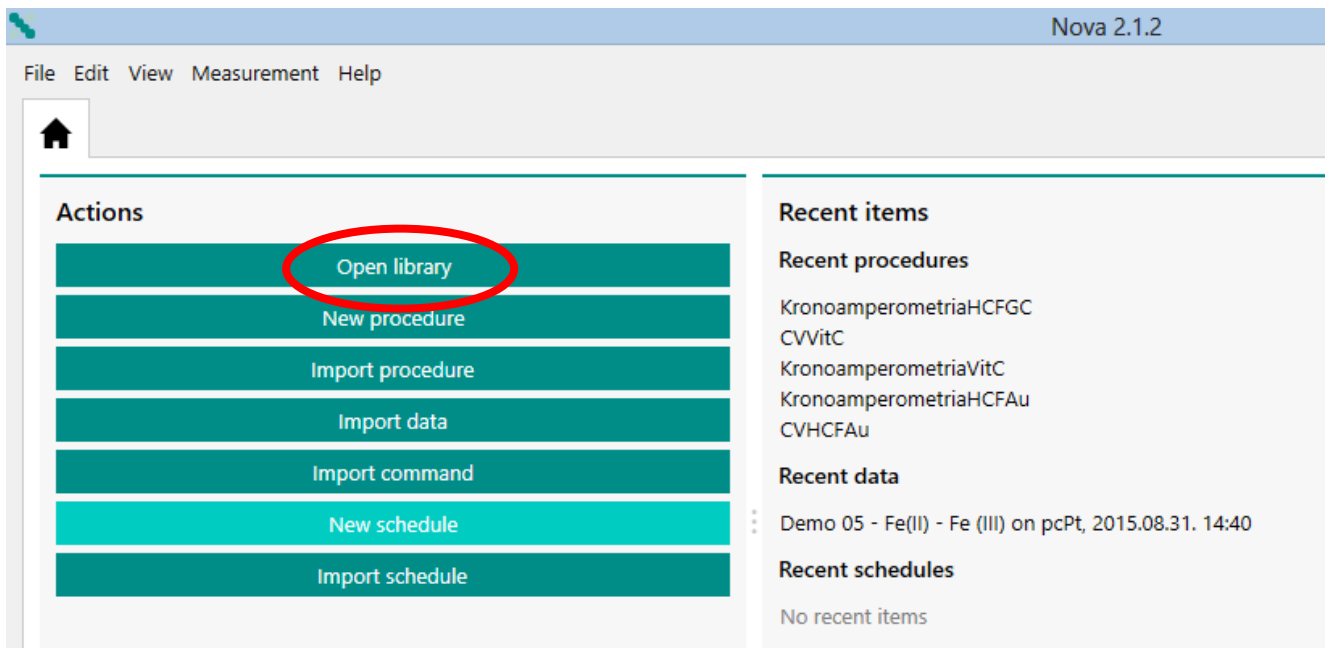
Choose the „Open library" menu by left-clicking on the mouse.
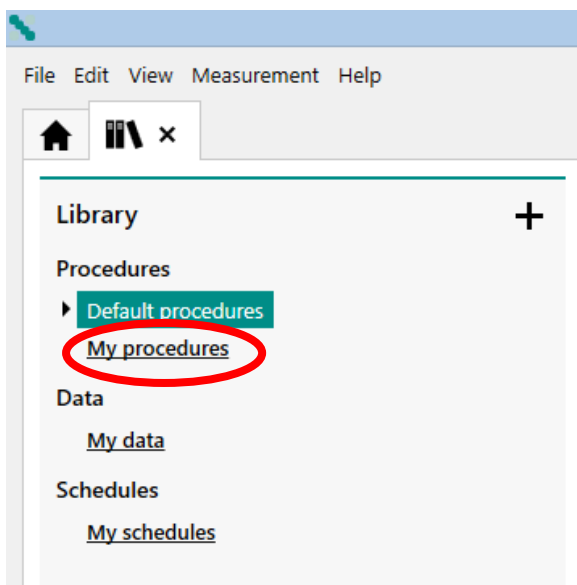
Clicking on the „My procedures" menu, you could find the procedures (programs) used in the measurements.
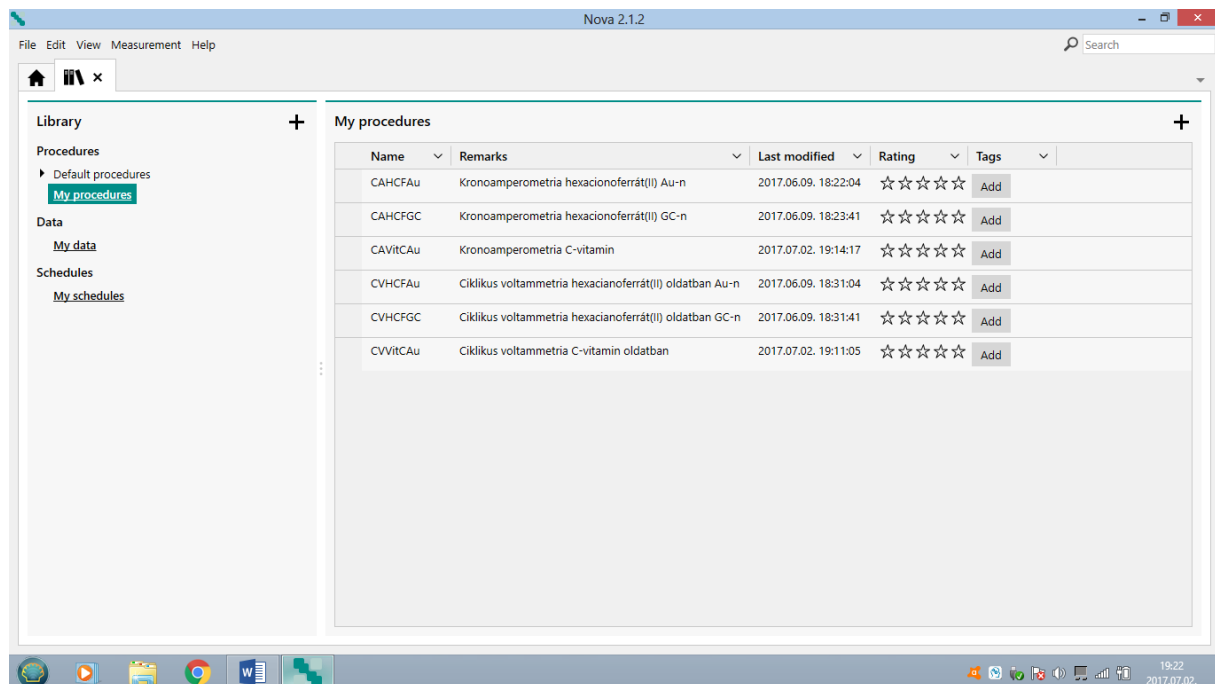
Choose the appropriate procedure to what you want to do. Read the „Remarks" field also to choose the right program.

For Chronoamperometric measurements, the parameters given in the measurement description are already set. The procedure looks like this.
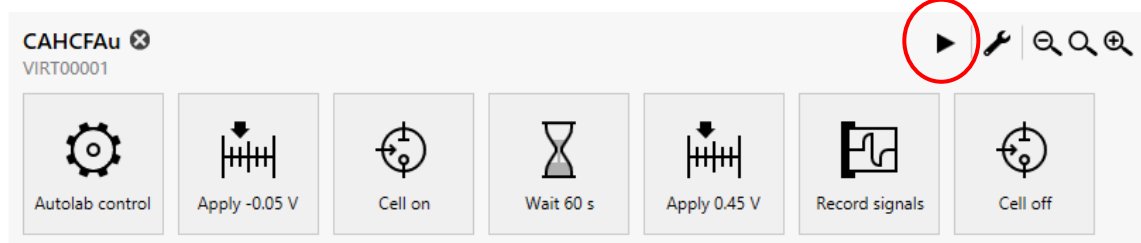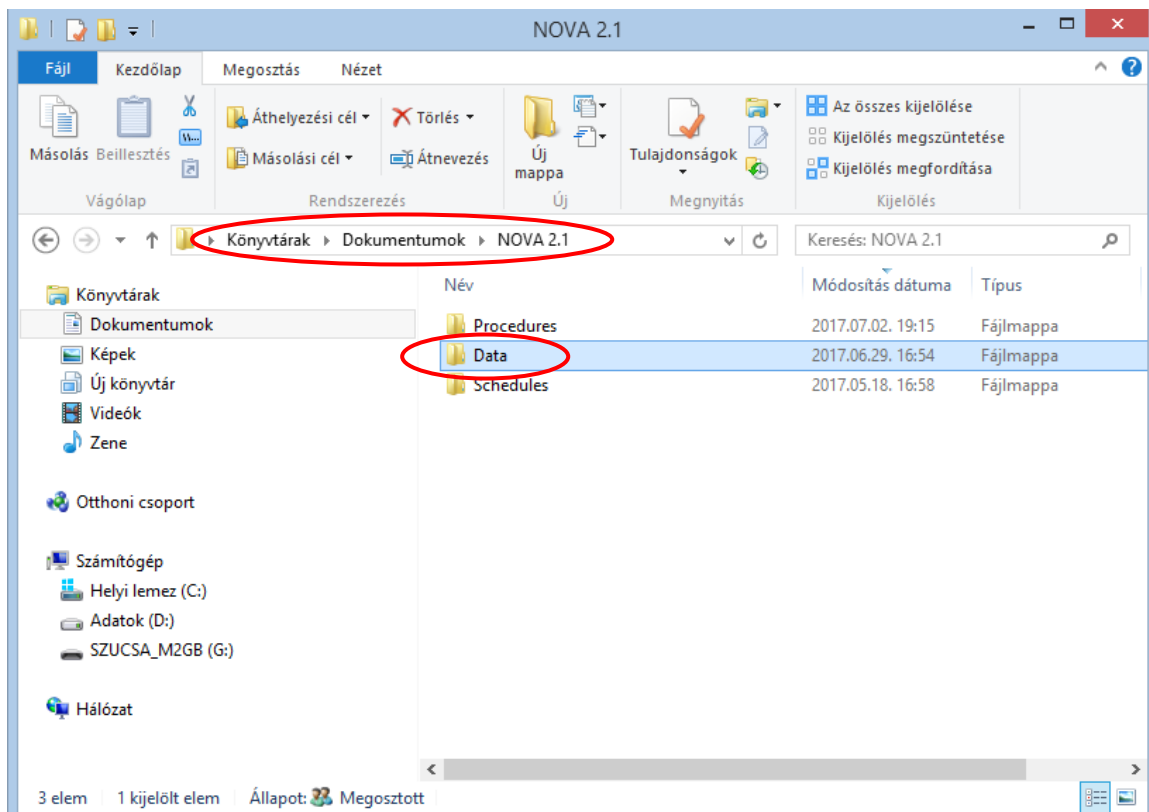

Each icon indicates "Commands", which makes the measurement happen. The first icon, "Autolab control" is used to detect the measuring instrument, to set the potentiostatic mode, and to select the current range. The second icon, "Apply", sets the initial electrode potential at the input of the potentiostat. The third icon, "Cell on", connects the potentiostat to the electrochemical cell. The fourth icon, "Wait", keeps the working electrode for the specified time at the initial potential. The fifth icon, "Apply", indicates the potential step to the specified value. The sixth icon, "Record signals", performs the measurement of the time depending current after the potential step. The seventh icon, "Cell Off" turns the cell off from the potentiostat, finishes the measurement. The measurement procedure and the measured data are automatically saved in a .nox file that can be found later on the "My data" menu in the "Library" menu. The data file name is the file name of the "Procedure" plus a "Timestamp", i.e. when the measurement was made. Therefore, it is important to note the order of measurements so that their results can easily be found later.

The measurement can be started by clicking on the "Run" command in the "Measurement" menu on the top of the screen or by clicking the Play (▶) icon above the Procedure.

During the measurement, instead of the Play icon, the Pause (‖) and Stop (■) icons will be displayed. If you notice a problem, stop the measurement.

During the measurement, the computer automatically plots the measured data in the Plots window in the lower left corner of the screen.

Physically, the data files are stored in the Dokumentumok Library in the NOVA 2.1 folder in the Data subfolder. From there, you can make a copy, e.g. to a pendrive.

„Behind" each Command icon you could find "Subcommands", shown on the right side of the Procedure pane, or you can see the details with the "More" command by clicking on the corresponding icon. If it is possible, do not change the parameters, or if this happened, DO NOT SAVE THE ALTERED PROCEDURE WITH ITS ORIGINAL NAME.

For cyclic voltammetric measurements, the "Procedure" looks like this:

Each icon indicates "Commands", which makes the measurement happen. The first icon, "Autolab control" (Autolab control) is used to detect the measuring instrument, to set the potentiostatic mode, and to select the current range. The second icon, "Message", asks for the applicable scan rate ("Scan rate (V/s)"). Once the measurement is started, it must be set to the desired value. The third icon, "Apply", sets the initial electrode potential at the input of the potentiostat. The fourth icon, "Cell on", switches the potentiostat to the electrochemical cell. The fifth icon, "Wait", keeps the working electrode for the specified time at the initial potential. The sixth icon, "CV staircase", represents the cyclic measurement between the lower and upper potential limits with the scan rate given in the „Message". The seventh icon, "Cell Off" turns the cell off from the potentiostat, finishes the measurement. The measurement procedure and the measured data are automatically saved in a .nox file that can be found later on the "My data" menu in the "Library" menu. The data file name is the file name of the "Procedure" plus a "Timestamp", i.e. when the measurement was made. Therefore, it is important to note the order of measurements so that their results can easily be found later.

The measurement can be started by clicking on the "Run" command in the "Measurement" menu on the top of the screen or by clicking the Play (▶) icon above the Procedure.

During the measurement, instead of the Play icon, the Pause (❚❚) and Stop (■) icons will be displayed. If you notice a problem, stop the measurement.

During the measurement, the computer automatically plots the measured data in the Plots window in the lower left corner of the screen.

Physically, the data files are stored in the Dokumentumok Library in the NOVA 2.1 folder in the Data subfolder. From there, you can make a copy, e.g. to a pendrive.

„Behind" each Command icon you could find "Subcommands", shown on the right side of the Procedure pane, or you can see the details with the "More" command by clicking on the corresponding icon. If it is possible, do not change the parameters, or if this happened, DO NOT SAVE THE ALTERED PROCEDURE WITH ITS ORIGINAL NAME.

The only parameter you have to set is "Scan rate (V/s)". For sequential runs, use the order that is described in the measurement description. (For example HCF solutions 0.320 V/s, 0.160 V/s, 0.080 V/s, etc.)

IMPORTANT! NOVA uses Windows default decimal separator. See that the "Message" contains the default value, and that the entered value is also correct! (For example, 0,08 or 0.08!) The data exported from the NOVA also uses the same decimal separator. If necessary, change it to the settings in the Excel Options menu in accordance with the Windows setup!
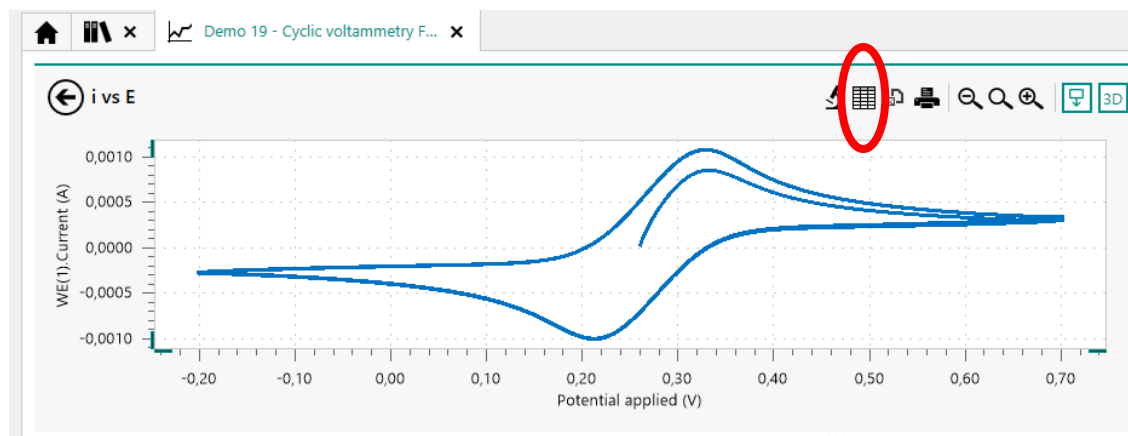
2. Description of evaluation (Autolab potentiometer and NOVA controller program)

The NOVA software used for the measurements can be applied for many purposes, but the User's manual is 1196 pages, which is a bit tiring in a lab to read. Therefore, only the most important functions are highlighted.

For the evaluation of voltammograms, it is necessary to determine the peak potentials (E_p), half peak current potentials ($E_{p/2}$) and the background corrected peak currents (I_p). This can be done in Excel after exporting the measurement data. To do this, double-click the "Plots" pane.
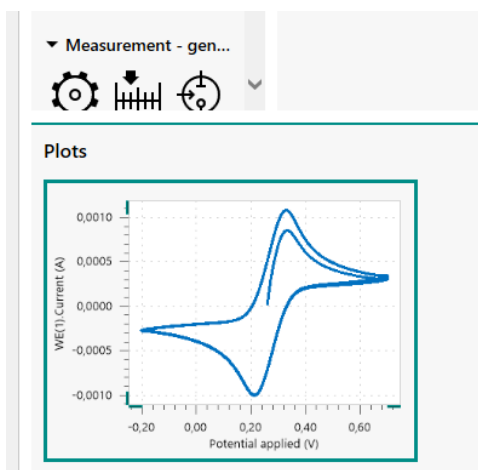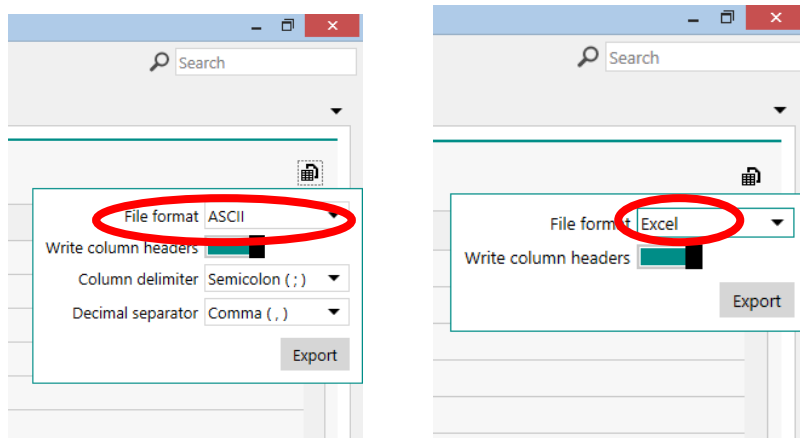

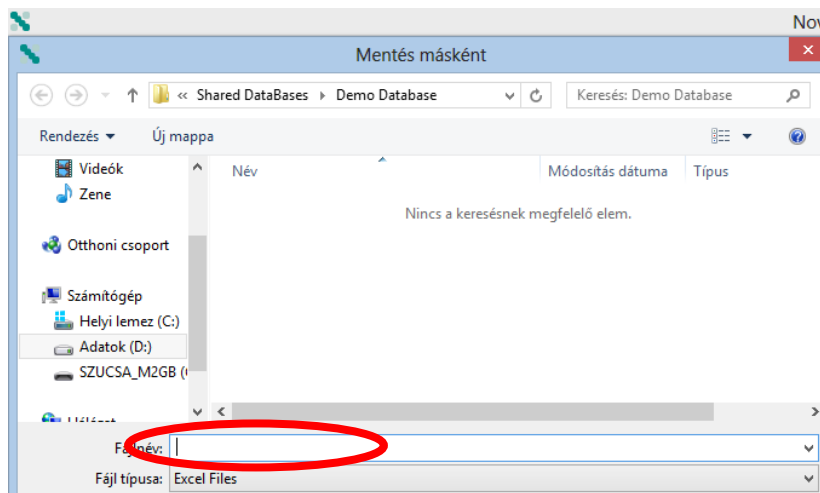Instead of the procedure, the measured plot is displayed in the main window.

Clicking on the „Show data" icon you could see a table.

In the top right corner there is a marker leading to the export function.

Select Excel Export from "File Format", and Windows Explorer will save the file to the desired name and location.


ATTENTION! The Excel file contains only the measured data (maybe calculated data, as well), not the description of the Procedure! It's still in the .nox files, do not delete them.

IMPORTANT! NOVA uses Windows default decimal separator. The data exported from the NOVA also uses the same decimal separator. If necessary, change it to the settings in the Excel Options menu in accordance with the Windows setup!

For Excel files, you can get the desired data, graphics, where needed.

For the evaluation of voltammograms, it is necessary to determine the peak potentials (E_p), half peak current potentials ($E_{p/2}$) and the background corrected peak currents (I_p). This can be done in Excel after exporting the measurement data, but (perhaps) simpler in NOVA with an "Analysis" function.
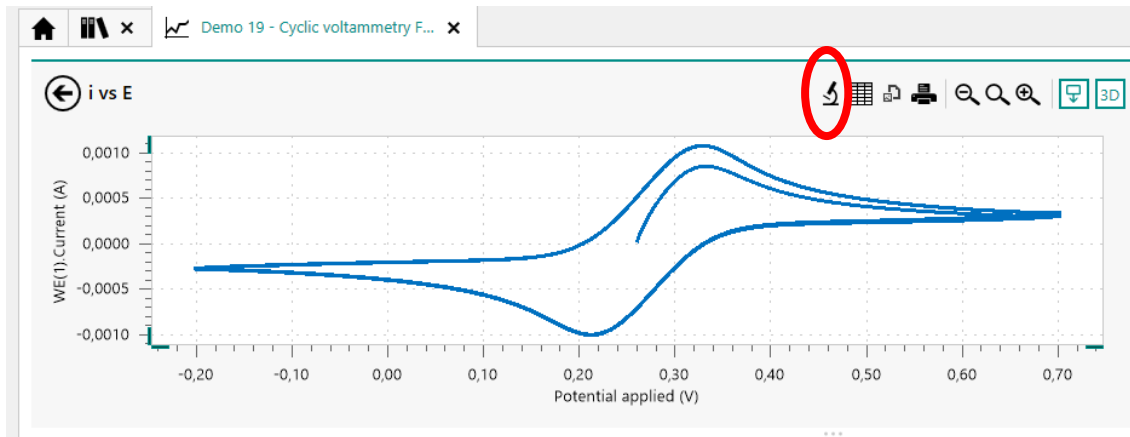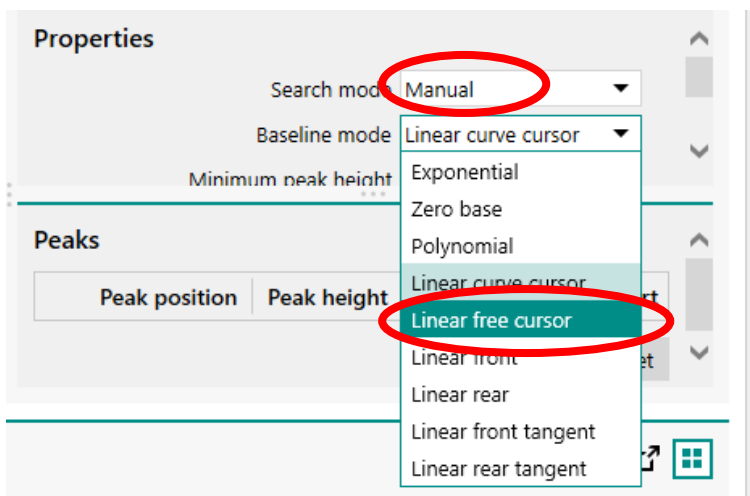
Click the microscope icon to see a menu. Select "Peak search" analysis.
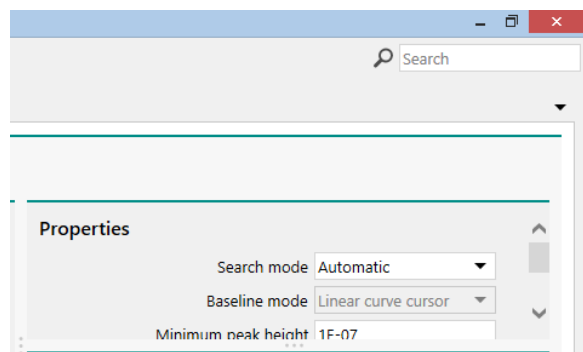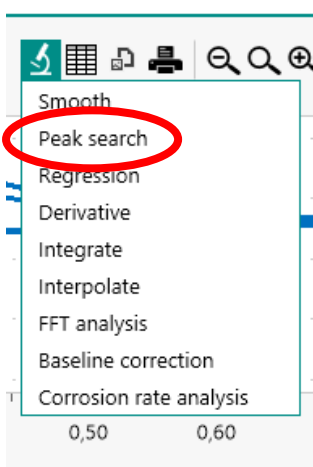
Then, a submenu "Properties" opens in the right window, which offers different versions of the peak search. The default setting is "Automatic", and the figure (or the table) also shows this result, but this is usually good for nothing!

Select "Manual", and "Linear free cursor" option.

Returning to the plot in the main window with the mouse, you could see a cross. Set this to the initial part of the voltammogram, and fix it with a click. Drag a straight line with the mouse that best fits the

first part of the voltammogram. The end of the line does not have to be on the curve (free cursor). You can fix the straight line with another click, and the results of the peak search appear in the "Properties" window on the right. (Another voltammogram is illustrated because there will be many peaks on the earlier example.)
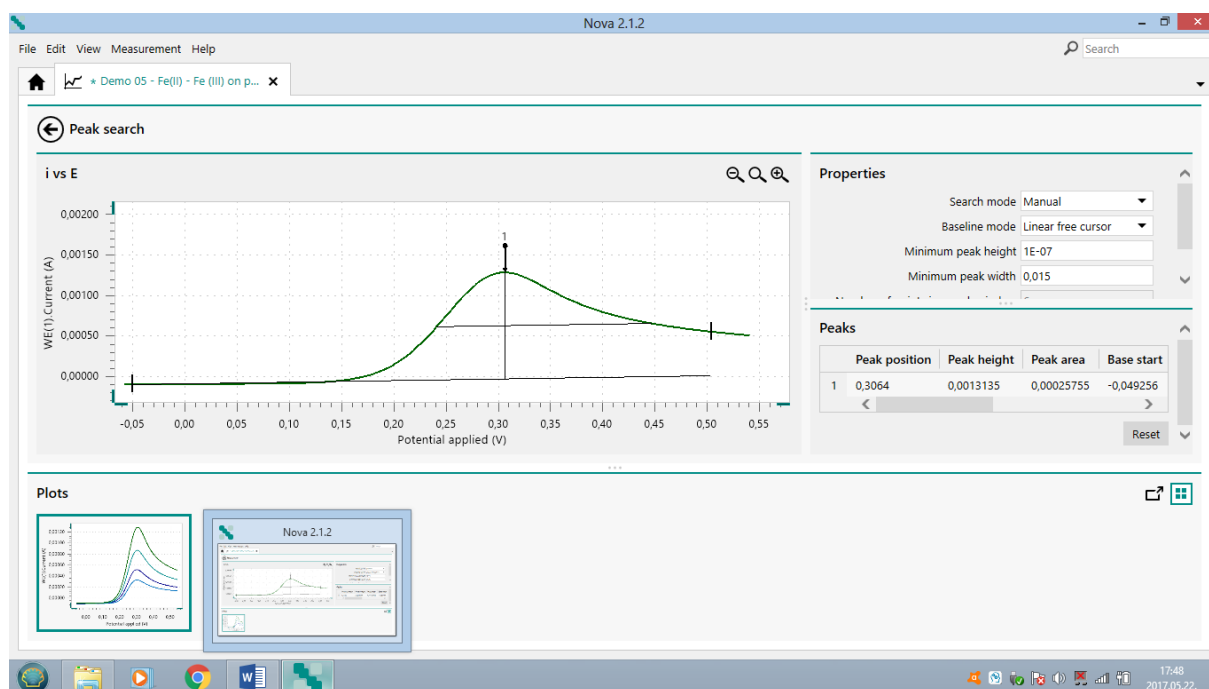
The same can also be done for the cathodic peak, however from the other end of the curve, the straight line must be fitted backwards.
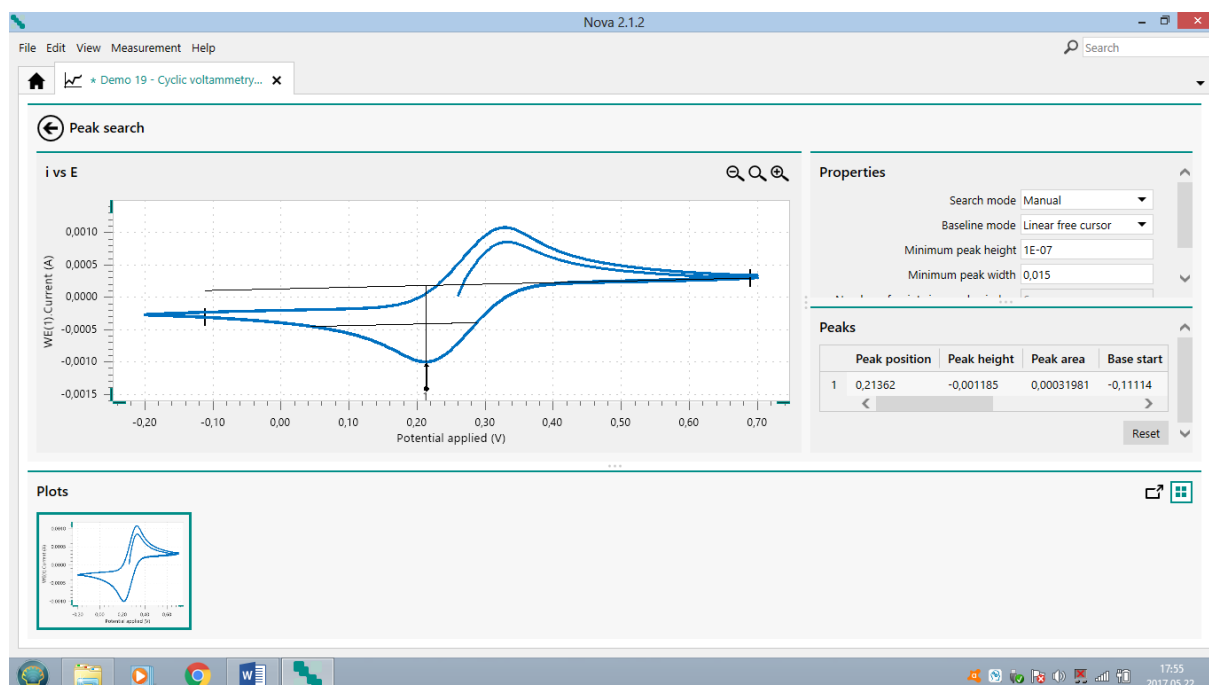
All of these can go in succession, the peaks are included in the right table ("Peaks"). If you are not satisfied with the fitting, you can resume the search at any time by pressing the "Reset" button.

Peaks

	Peak position	Peak height	Peak area	Base start	Base end	Peak width half height	Peak (1/2)	Peak sum of derivatives
1	0,21362	-0,001185	0,00031981	-0,11114	0,68977	0,2451	-0,07446	0,017343

Reset

The values in the table are:

1 Number of found peaks

"Peak position" is the electrode potential of the found peak in Volt

"Peak height" is the corrected value of the peak current in Amper

"Peak (1/2)" is the difference between the potential at the peak current and the potential at the half peak current, i.e. Peak (1/2) = $E_p - E_{P/2}$. (Signed!) (The other columns are not interesting now!)

	Peak position	Peak height	Peak area	Base start	Base end	Peak width half height	Peak (1/2)	Peak sum of derivatives
1	0,33325	0,00059343	8,8522E-05	0,26978	0,69458	0,12473	0,045121	0,023525
2	0,21362	-0,0010552	0,00023433	-0,20142	0,40405	0,18536	-0,072126	0,017343
3	0,32593	0,0010691	0,00021897	0,14038	0,69946	0,17293	0,068819	0,018198
4	0,21362	-0,00106	0,00023585	-0,20142	0,40405	0,18559	-0,072416	0,01737
5	0,32593	0,0010701	0,00021903	0,14038	0,69946	0,17282	0,068775	0,018354
6	0,21362	-0,0010615	0,00023618	-0,20142	0,40405	0,18554	-0,072407	0,017334

Data can not be exported directly, but when the data is marked with the mouse you can use Ctrl-C to put those on the clipboard, and then use the command Ctrl-V, for example, to copy those to Excel. Only the data, the header will not pass! Afterwards, you can delete unnecessary parts.

IMPORTANT! NOVA uses Windows default decimal separator. The data exported (or copied) from NOVA also uses the same decimal separator. If necessary, change it to the settings in the Excel Options menu in accordance with the Windows setup!
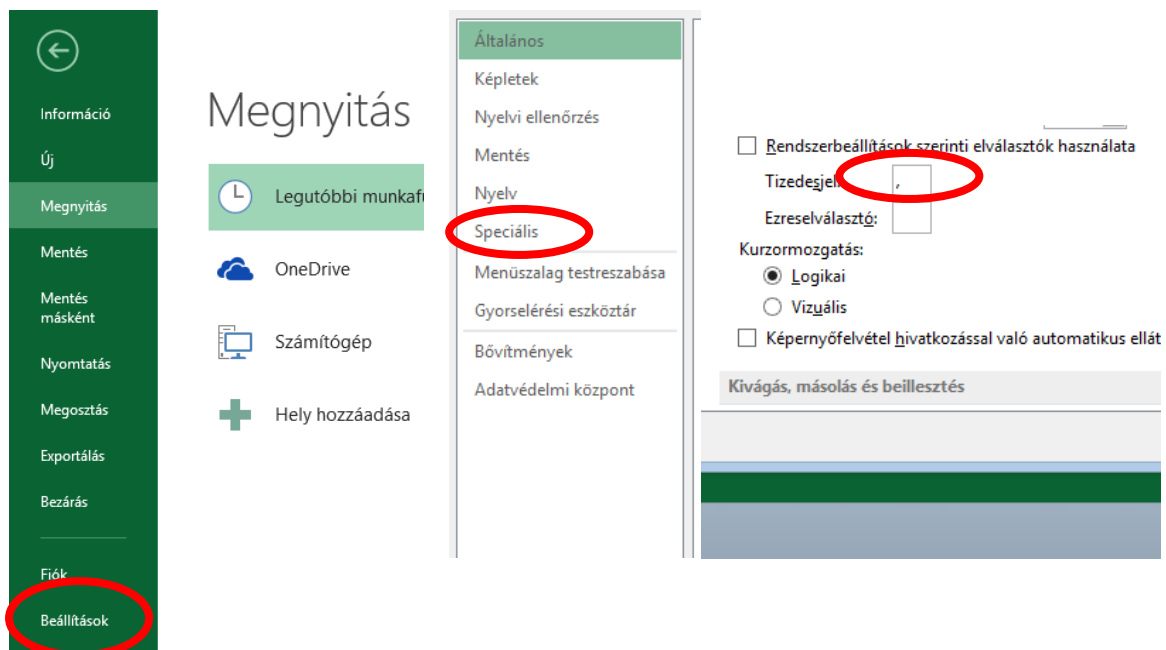
For chronoamperometric measurements it is recommended to integrate (calculate the charge-time function) in NOVA (you could spare an Excel programming). In the "Plot" window, call up the "Integrate" analysis command!
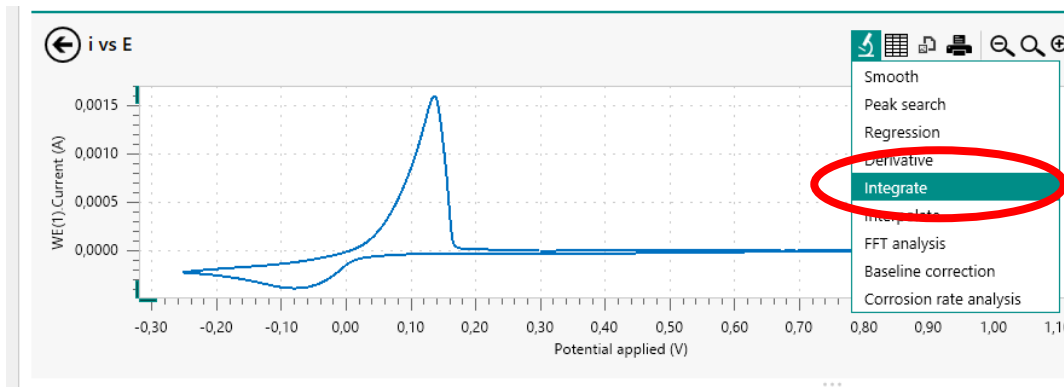
The result is a new figure and the extension of the data file with integrated values.

WE(1).Current (A)	Scan	WE(1).Potential (V)	Index	Q+	Q-	Integrated WE(1).Current (A V)	Current range
-6,32935E-7	1	0,300781	1			0	1 mA
1,02966E-6	1	0,303375	2			4,84268E-10	1 mA
2,14844E-6	1	0,304962	3			4,36381E-9	1 mA
2,87384E-6	1	0,306824	4			1,04945E-8	1 mA
3,14392E-6	1	0,311951	5			1,78404E-8	1 mA
3,15582E-6	1	0,312744	6			2,55305E-8	1 mA
3,61328E-6	1	0,313965	7			3,37936E-8	1 mA

Exporting the measurement results to Excel will include the measured currents and the calculated charge as well.

The rest are just time, patience and lots of work, and the evaluation is ready!
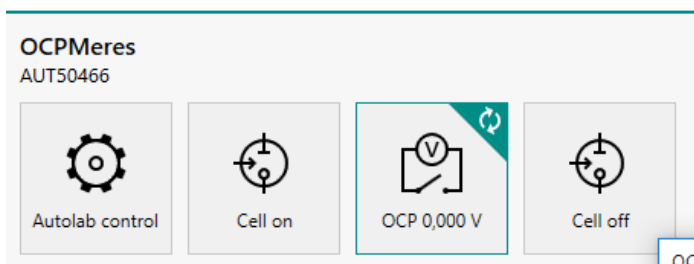
14. Chronoamperometry

1. Description of the measurements

**(Autolab potentiostat and NOVA controller program)**

IMPORTANT SUPPLEMENT

After each electrode exchange and / or solution change, the first thing is a cell control!
To do this, select "OCPMeasurement" from the "My procedures" list. The procedure looks like this.

The first icon, "Autolab control", is used to detect the measuring instrument and to set the potentiostatic mode. The second icon, "Cell on", connects the potentiostat to the electrochemical cell. The third icon "OCP" (Open Circuit Potential) begins to measure the potential difference between the working and reference electrodes (OCP) when no current flows (the electric circuit is open between the working and the counter electrode). The fourth icon "Cell Off" turns the cell off the potentiostat, finishes the measurement.
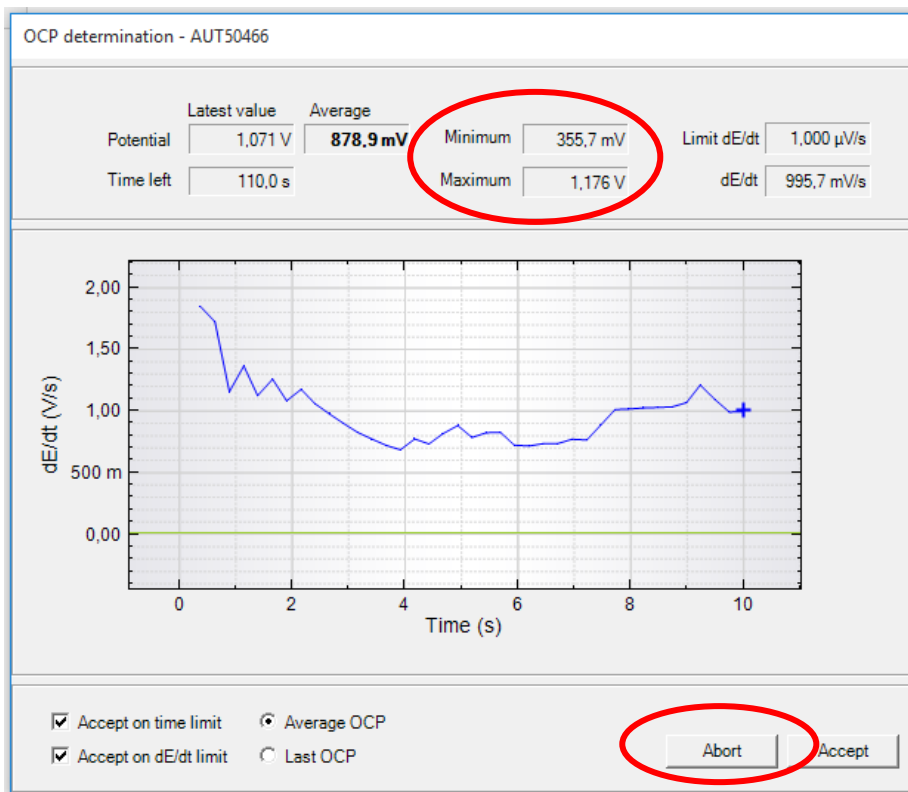
If, after starting the measurement, it is found that the difference between the Minimum and Maximum Potential is too high, or the blue line indicating the time dependence of the time derivative of the electrode potential (dE/dt) shows too high values (V/s and not µV/s), interrupt the measurement with the "Abort" command. **THERE IS SOME TROUBLE!**

It is likely that the working and / or the reference electrode is not properly connected, the system can not be used! (The system did not damage yet because there was no current during the measurement!) **Check the connections and run the procedure again.**

R (blue) reference electrode (NCE), C (black) counter (auxiliary) electrode (Pt), W (1,2) (red, plugged together) working electrode (Au or GC). There is a green ground, you do not have to use it, but do not touch any electric conductor with it.
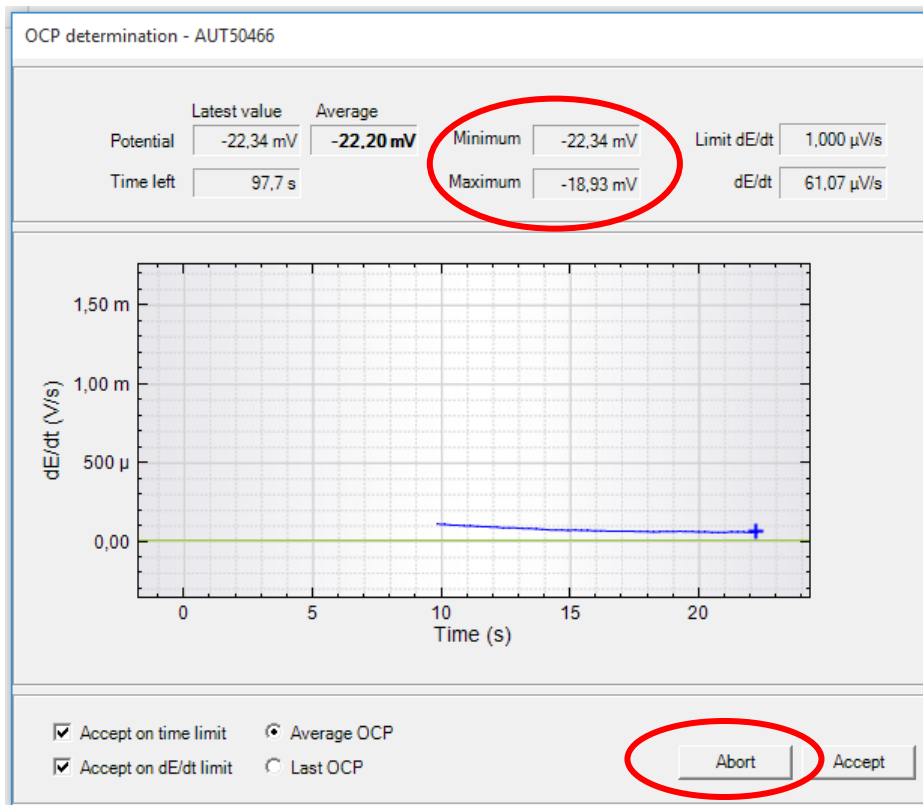
If, after starting the measurement, it is found that the deviation between the Minimum and Maximum Potential is small max. 10-20 mV, and the blue line indicating the time dependence of the time derivative of the electrode potential (dE/dt) shows normal (mV/s or µV/s) values, you can interrupt the measurement with the "Abort" command. **The cell connection is OK!**

The measurement procedure and the measured data are automatically saved in a .nox file that can be found later in the "My data" menu in the "Library" menu. The data file name is called "OCPMeasurement" plus a "Timestamp", i.e. when the measurement was made. These files should normally not be used at the time of evaluation, but only for diagnostic purposes.