Getting Started with EC-Lab®:
CV: Cyclic Voltammetry

AIM

The aim of this presentation is to guide you to set the appropriate parameters to perform a CV measurement.

Only CV technique will be discussed hereafter but the information given in the presentation can be adapted to CVA, LP, MP techniques.

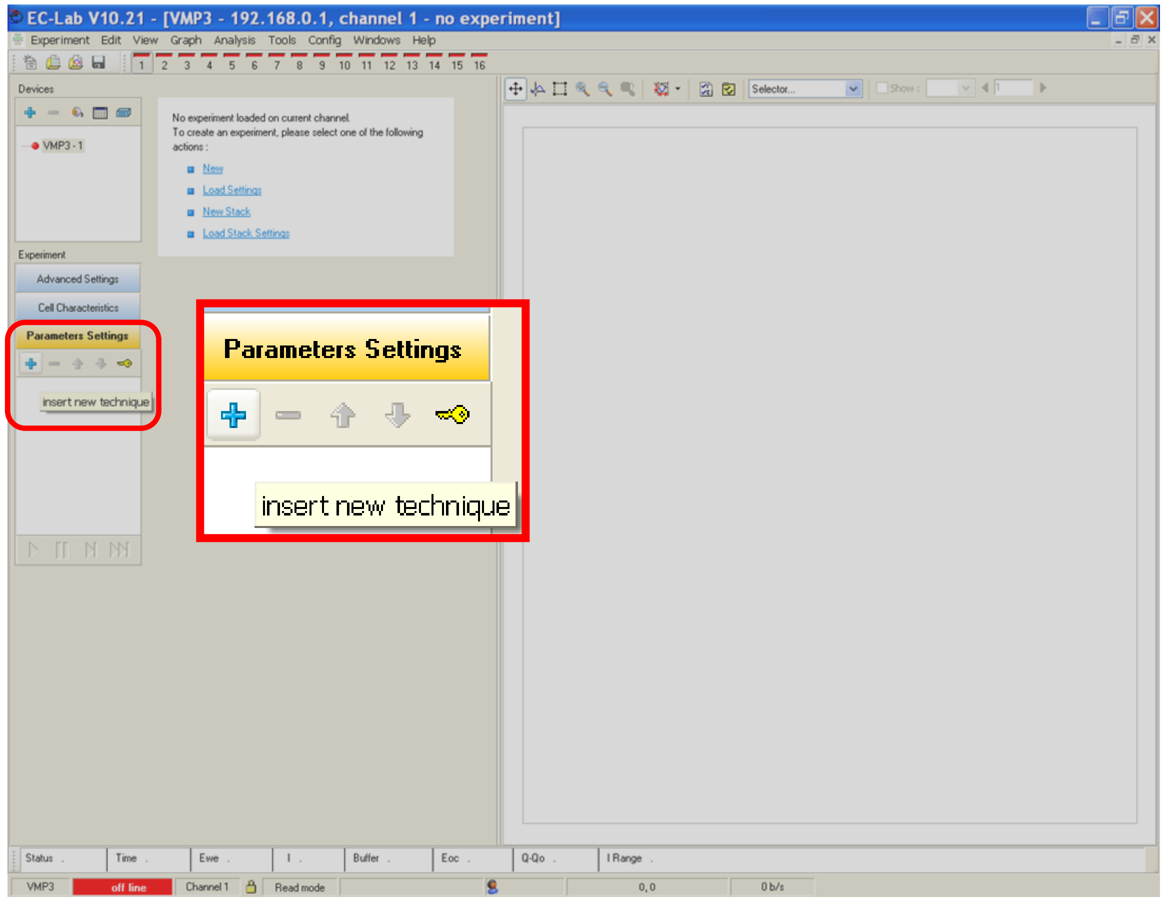
PROCEDURE

1. Insert the CV technique
2. Insert other(s) technique(s) if needed
3. Set the « Advanced Setting » tab
4. Set the « Cell Characteristics » tab
5. Set CV technique
6. Start the experiment

NOTE:
It is assumed that the computer and the instruments are connected. This is explained in the Getting Started named "EC-Lab®: Connection to the instrument(s) & Channel(s) selection"

1- Insert the CV technique

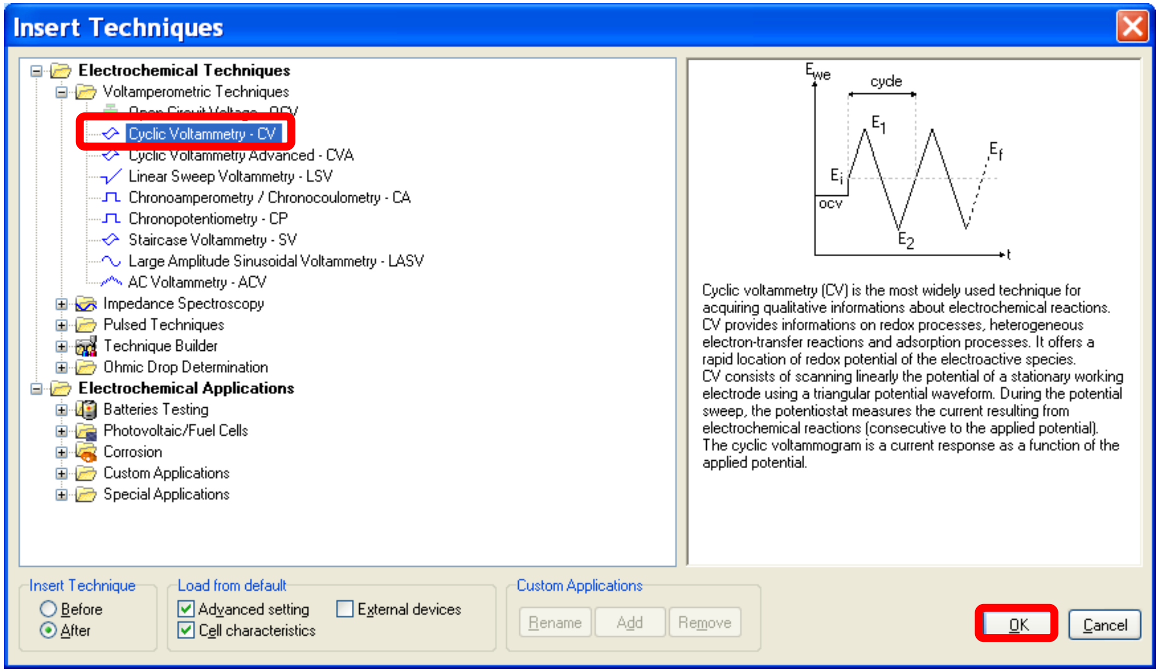
- In the experiment frame, click on the  button to insert the new technique



1- Insert the CV technique

- Select "Cyclic Voltammetry – CV" technique available in the "Voltamperometric Techniques" folder. The technique is highlighted in blue when selected

- Click on the "OK" button

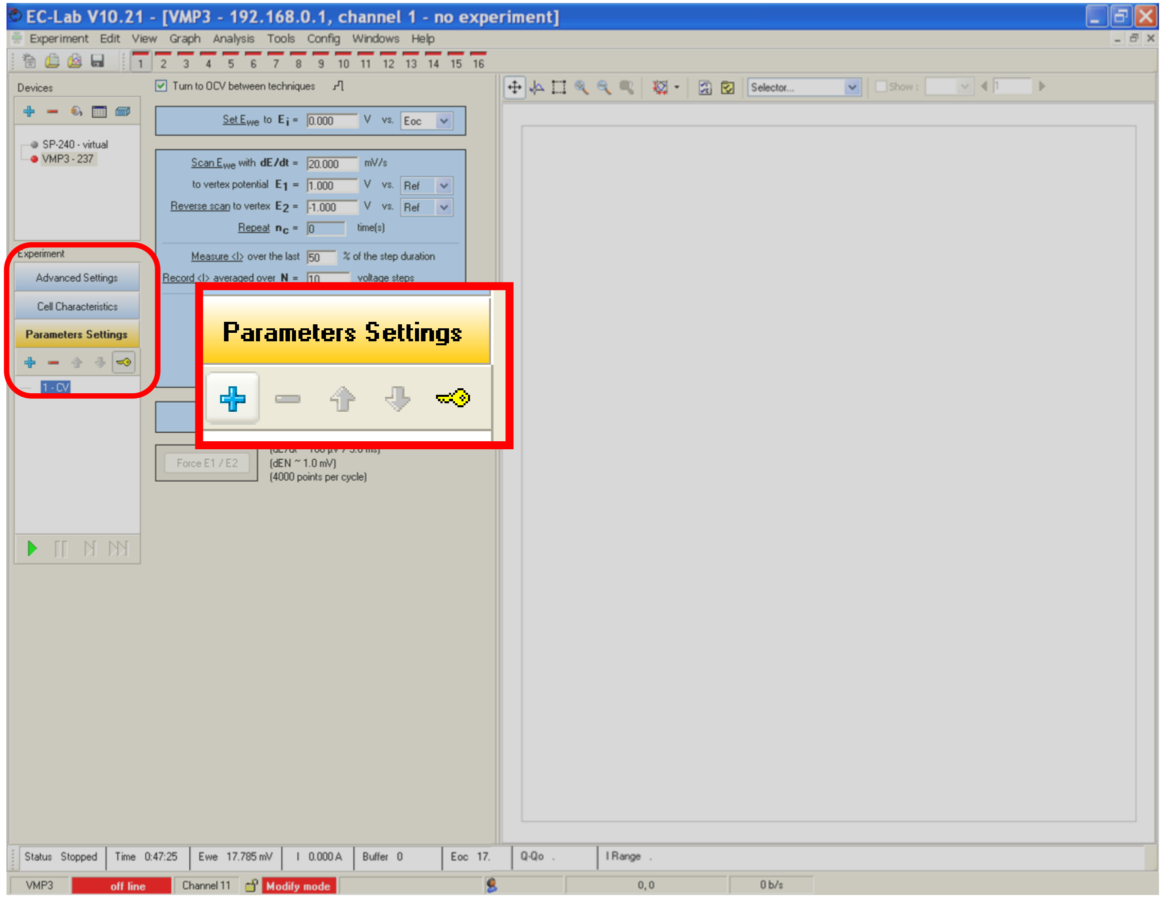


2- Insert other(s) technique(s)

• Click on the button to insert any additional technique(s).

Note: it is possible to remove a technique or to move a technique before or after. First select the technique that you want to remove/move (the technique will be highlighted in blue) and then click on the appropriate button.

- To add
- To remove
- To move before
- To move after



3- Set the « Advanced Setting » tab

- Click on the **Advanced Settings** tab.

  Then the « Advanced Settings » window is shown.

  The most important parameters to set for CV experiment is the « Filtering » and « Ultra Low current » correction block, only available for SP-300 family.
  Data can be filtered afterwards by software.

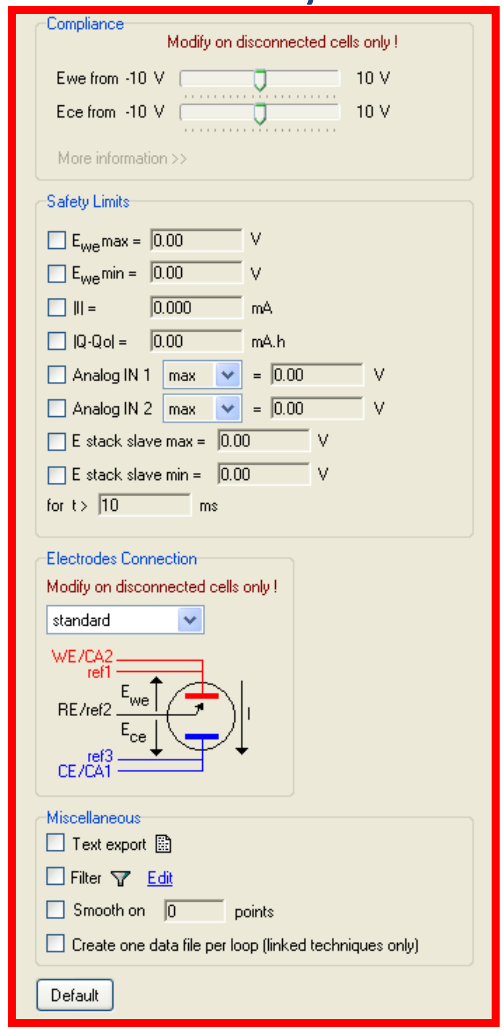
NOTE:

This window is different for the VMP3 family and for the SP-300 family.

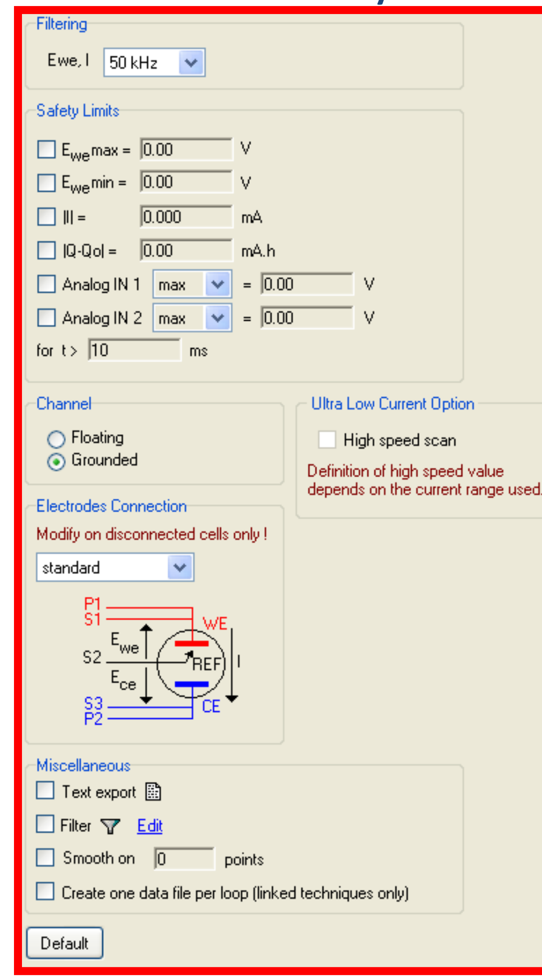
VMP3 family:
SP-50, SP-150, VSP, VMP3, CLB-500, CLB-2000, HCP-803, HCP-1005

SP-300 family:
SP-200, SP-240, SP-300, VSP-300

VMP3 family

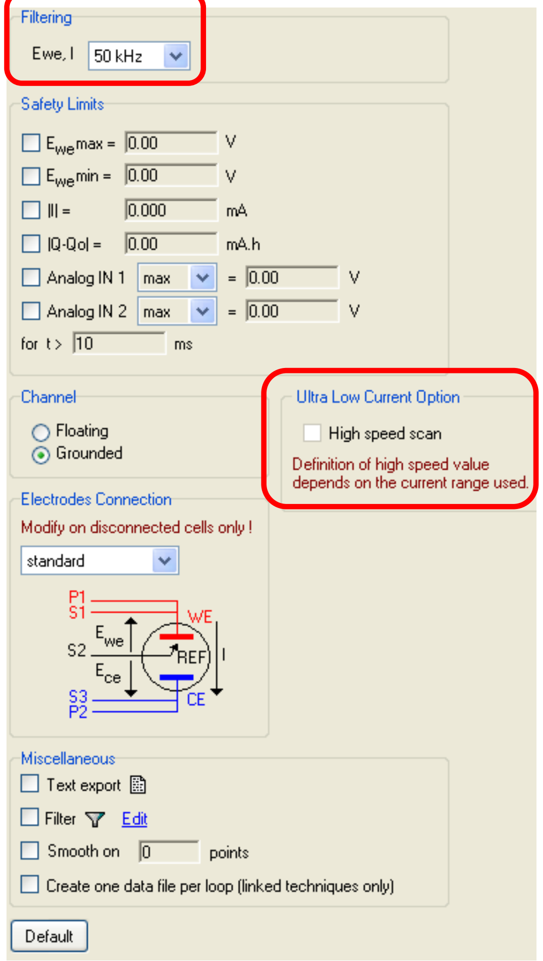


SP-300 family



3- Set the « Advanced Setting » tab

SP-300 family



Filtering

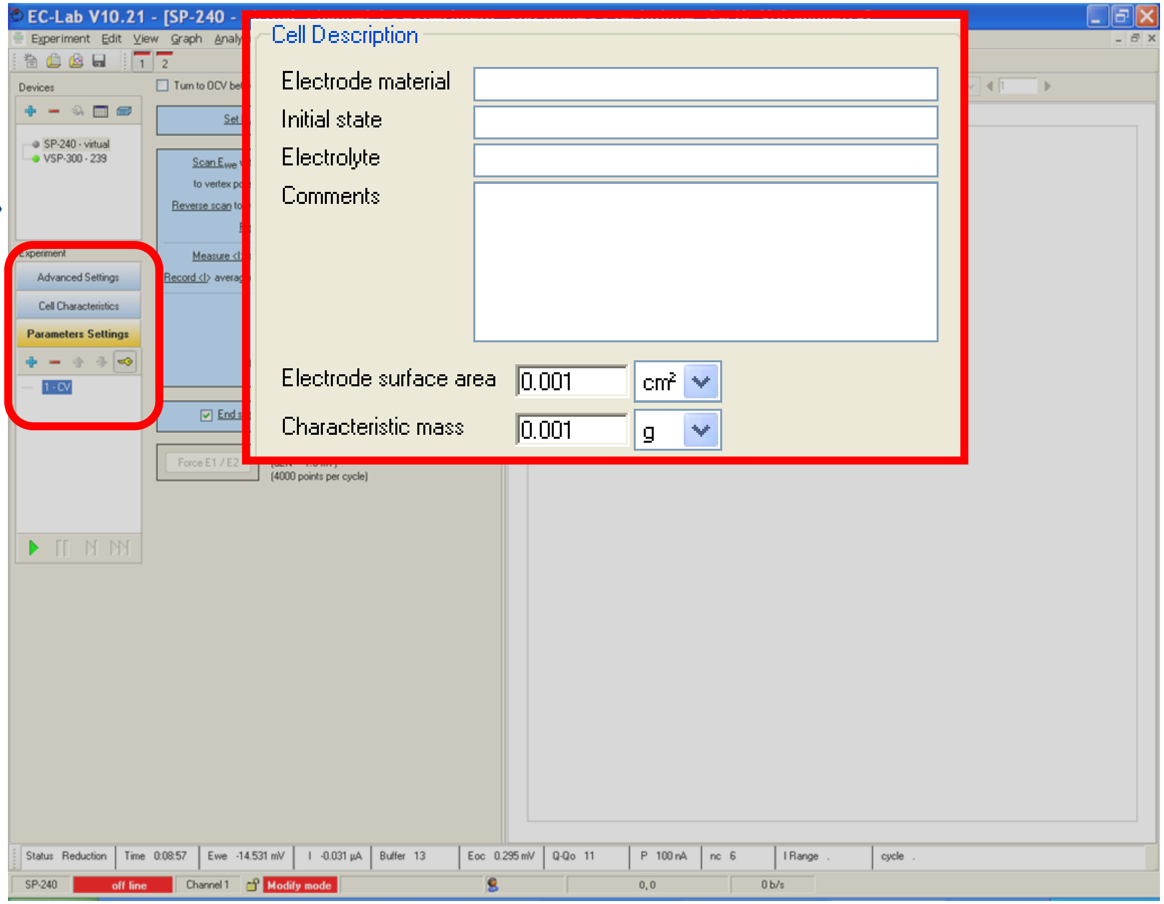
Three analog filters are offered: 50 kHz, 1 kHz and 5 Hz.
Select the 5Hz-filter to remove the aliasing from external perturbation (50 or 60 Hz depending on the country).

Ultra Low current (only available if ULC cable is connected to the potentiostat)
This option has to be ticked if the user works at very low level of current and at high scan rate.

4- Set the « Cell Characteristics » tab

- Click on the **Cell Characteristics** tab.

  Then the « Cell Characteristics » window is displayed.



- Information about the cell and some comments.

- Electrode surface area has to be set if the user want to work with current density (mA/cm^2) instead of current (mA).

NOTE:
All these infomation are stored in the data file

5- Set the CV technique

- Click on the **Parameters Settings** tab or directly on the technique CV in the list of technique. The technique is highlighted in blue.

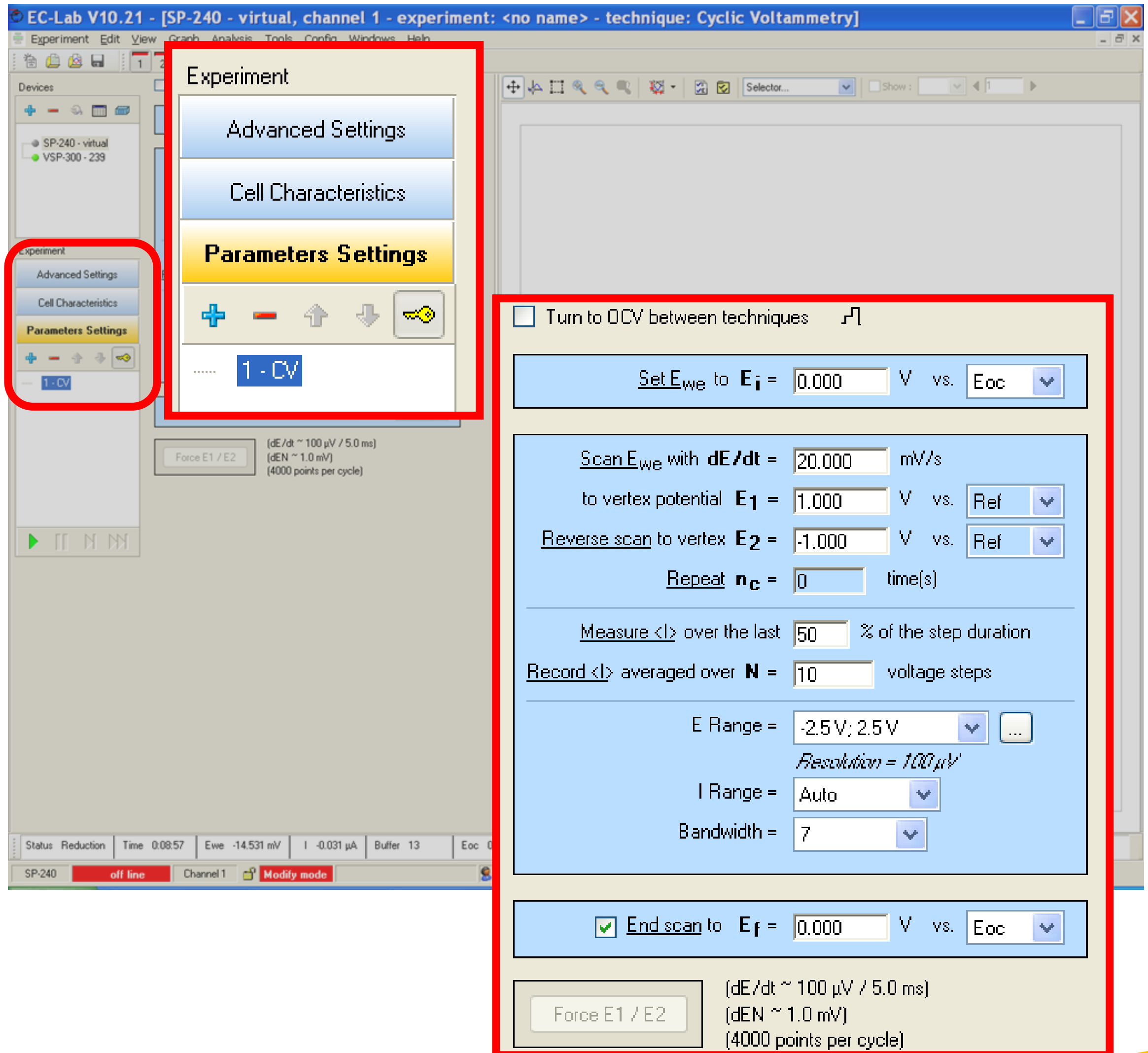
- Three different blocks compose the CV technique (described in the next slides):
- In the first block , the initial potential is defined.
- In the second block, the voltage sweep and the recording conditions are defined.
- In the third block, the final potential is defined. It can be deactivated

TIP:

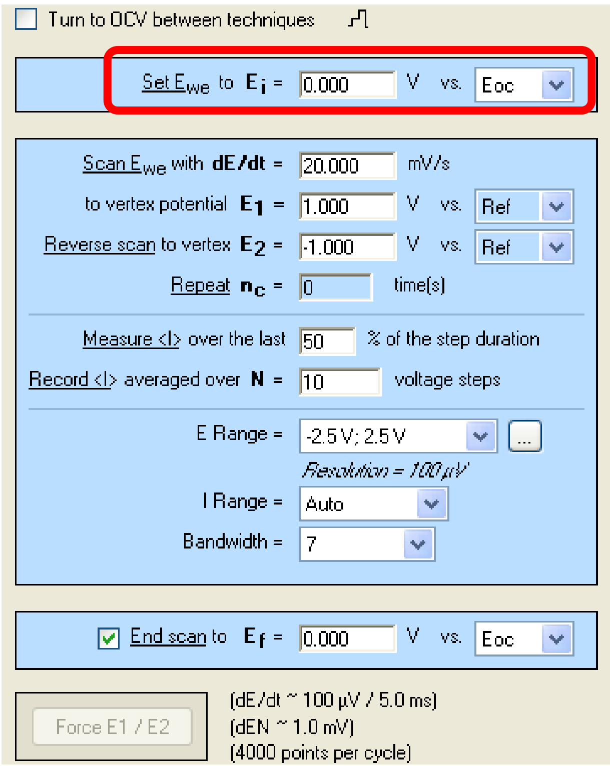
If the user would like to set a conditioning period before starting the voltage ramp, he has to set a CA technique before the CV or use the CVA technique.

NOTE:

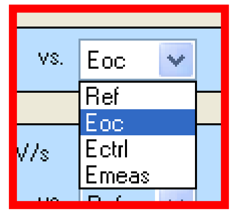
All the settings may be changed during the experiment (except Irange, Erange, bandwidth).



5- Set the CV technique



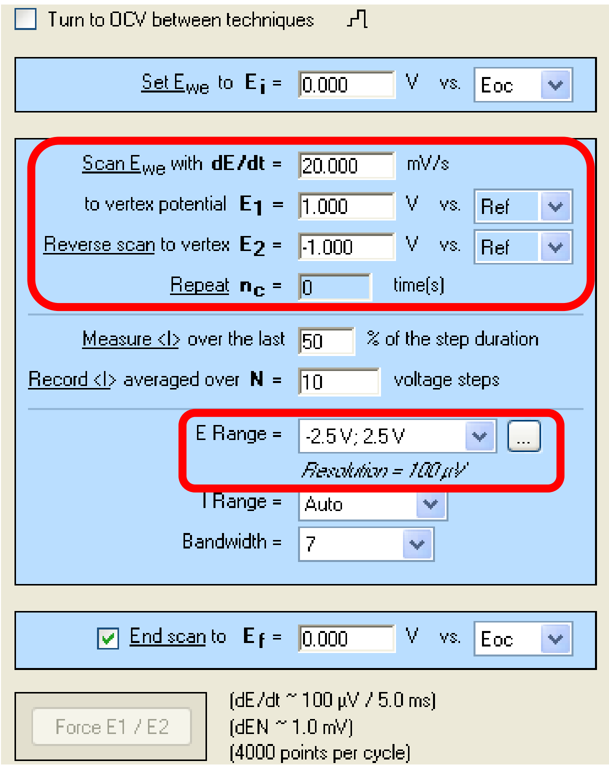
- Defines the potential at which the voltage sweep starts. This can be defined *versus* several voltage reference.



Ref (the potential of the reference electrode)
Eoc (Open circuit voltage)
Ectrl (potential of the previous controlled voltage, if a technique is set before the CV)
Emeas (potential of the previous measured voltage, if a technique is set before the CV)

TIP:
Set 0 V vs **Eoc** to avoid a current jump at the beginning of the CV.

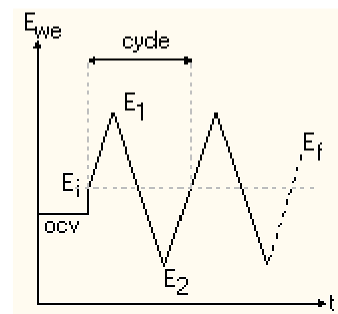
5- Set the CV technique



- Defines the scan rate and vertex potentials (E_1 and E_2). It is possible to define vertex potential versus several references (more info in the previous slide).

  The voltage Ei, E1, E2 Ef have to be in the Erange. The latter can be modified by or buttons. [-2.5; 2.5] V range is adapted to the electroactivity window of most of the electrolytes.

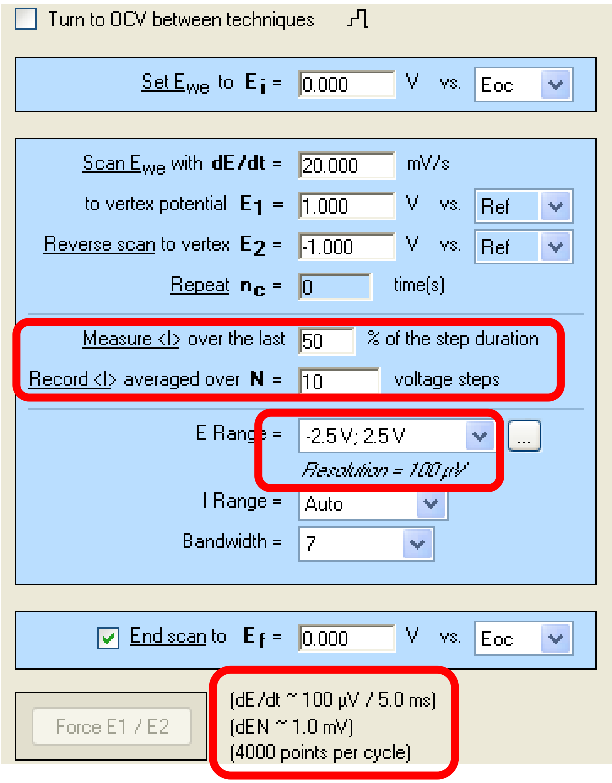
  It is also possible to cycle between E_1 and E_2 several times. This is the « Repeat » box n_c.



TIP:
Better to set E_1 and E_2 vs Eref, OCV may change whereas Eref is absolute potential values .

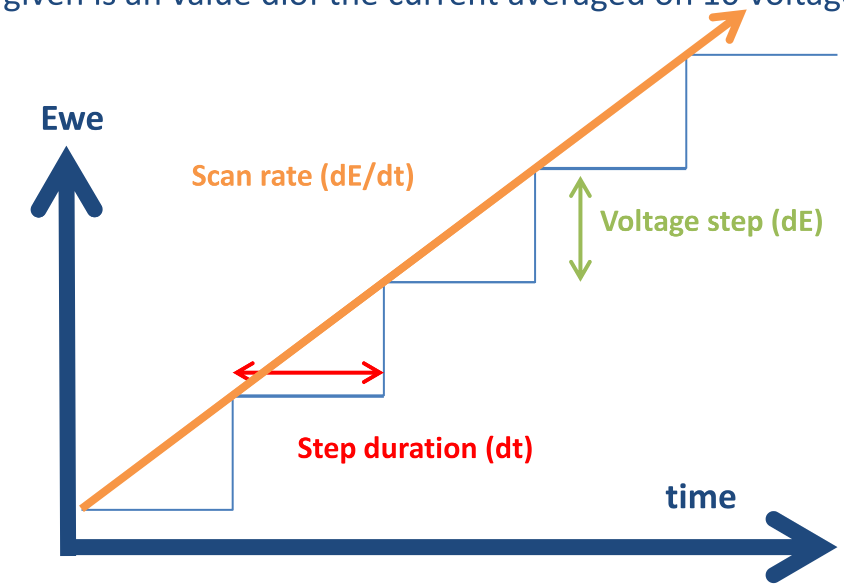
5- Set the CV technique

- defines how the instrument samples during the CV technique.



The first parameter define when the current is measured for each voltage step. Actually, to perform a ramp of potential, the potentiostat applies several voltage steps (discrete behavior, not analog).

The second one allows you to get a smoother data, it is recommended to use N = 10. Current given is an value diof the current averaged on 10 voltage steps.

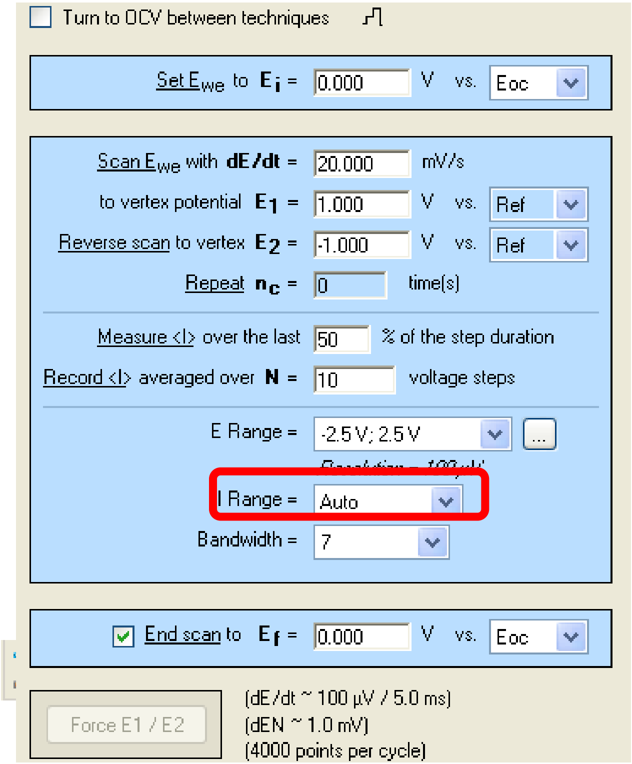


TIP:

The dE (step voltage) is dependent on potential resolution which is dependent on the Erange. So to get an optimized CV, Erange has to be as narrow as possible.

For information, the actual step duration (dt) and voltage step (dE) are indicated at the bottom of the technique.

5- Set the CV technique



Irange is the range of expected current.
It is possible to set autorange.

TIP:
For high scan rate. It is recommended to set a fixed Irange because the Irange shift duration may be no more negligible.

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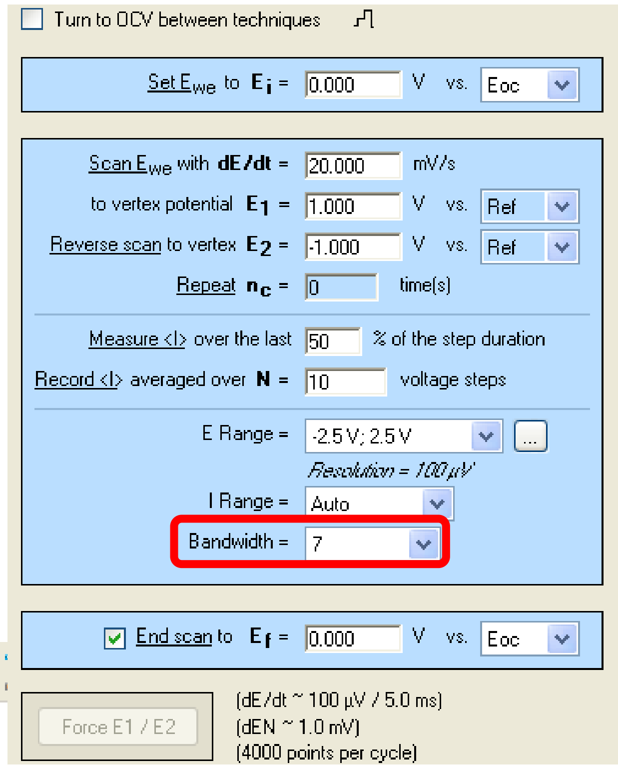
5- Set the CV technique



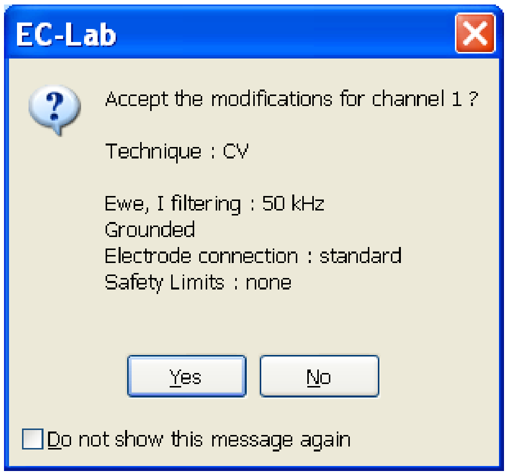
• defines the stability/speed of the instrument. Set fast bandwidth for high scan rate above 200 mV/s. Bd 7 for VMP3 family and Bd 9 for SP-300 family.
(See manuals and application notes for more information on the bandwidths).

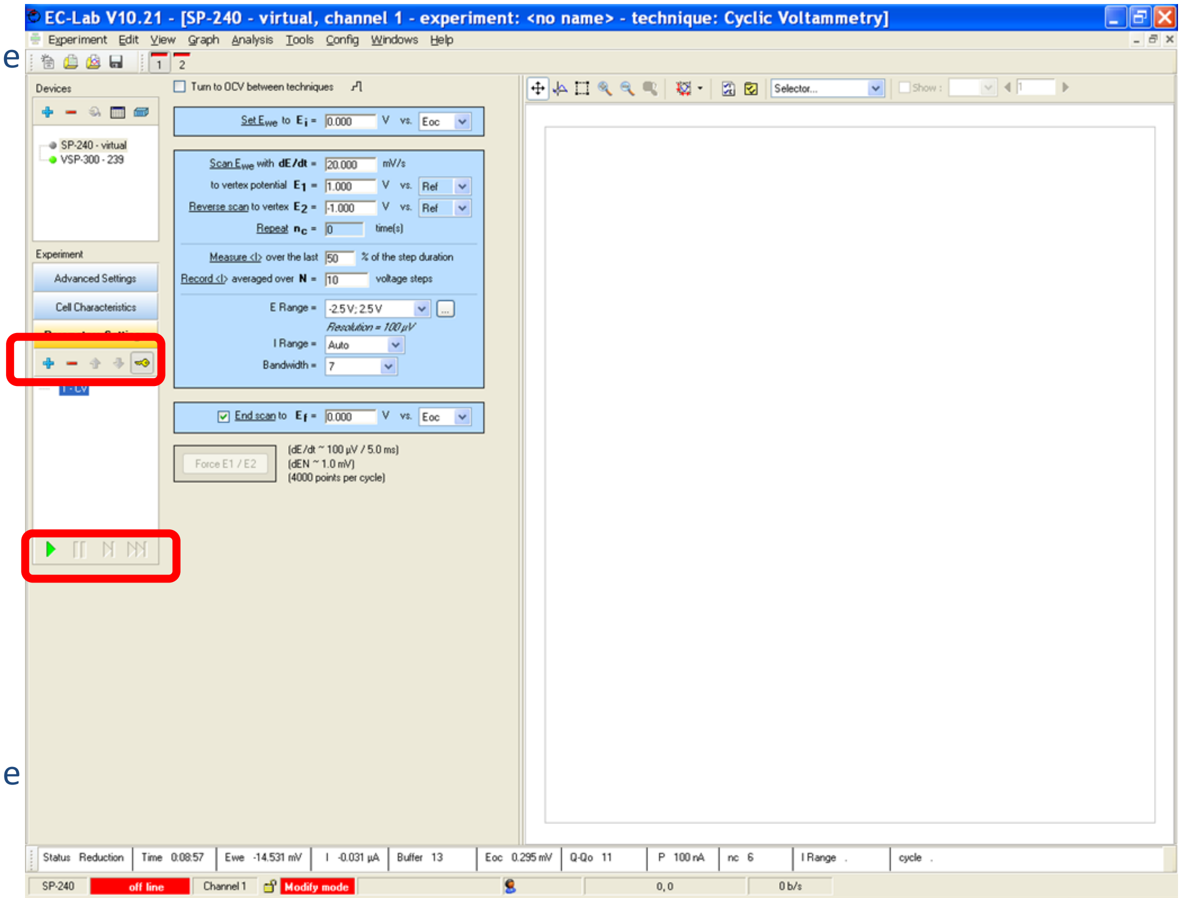
NOTE:
the bandwidth of the VMP3 family and the bandwidth of SP-300 family are not identical. Bandwidth 7 of VMP3 is different from the bandwidth 7 of the SP-300.

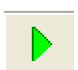
6- Start the experiment

- Click on  button to check if the settings are accepted

- Some warning messages may come up.

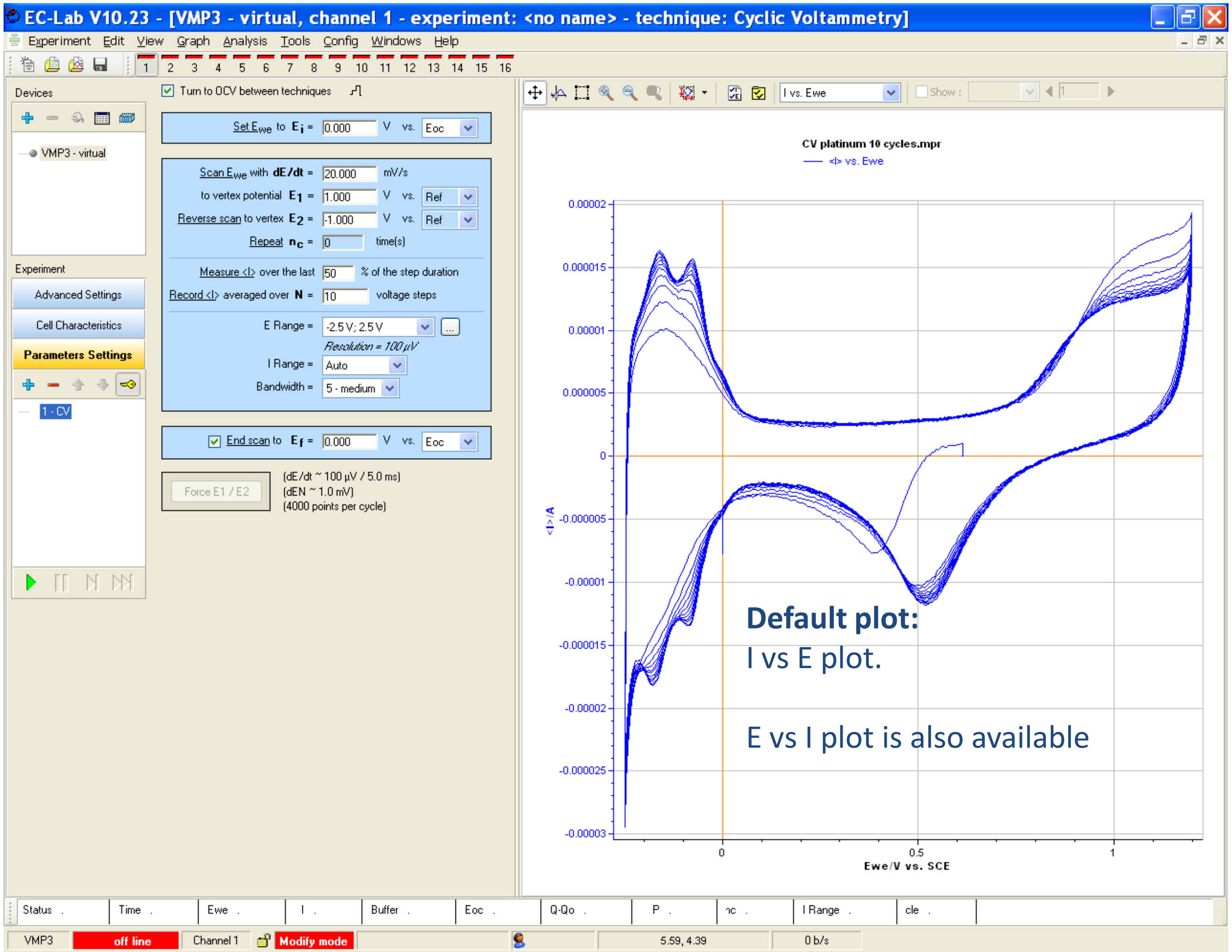




- Click on the  button to start the experiment.

- It is possible to stop, pause, go to next sequence, go to next technique by clicking on the buttons  .

Example of resulting data



Default plot:

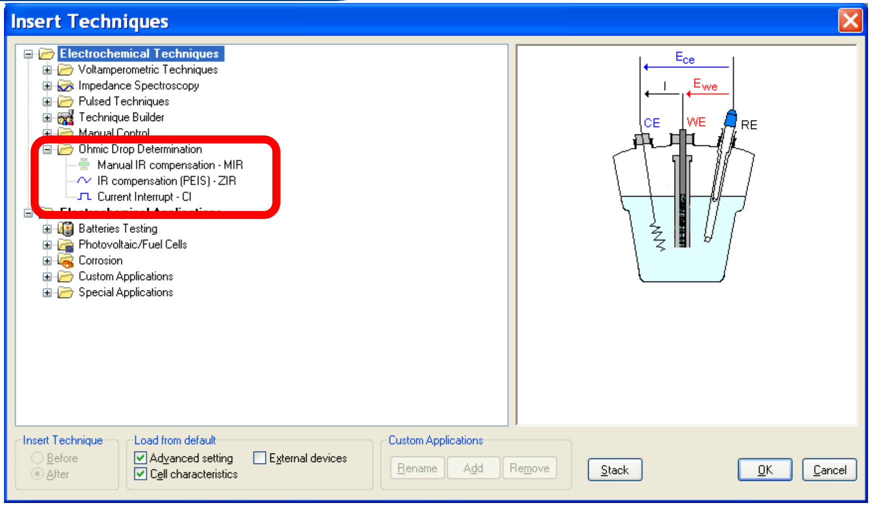
I vs E plot.

E vs I plot is also available

Few additional tips

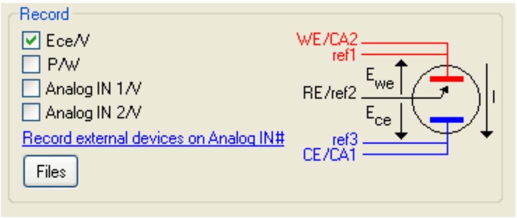
- **Ohmic drop** can be compensated.

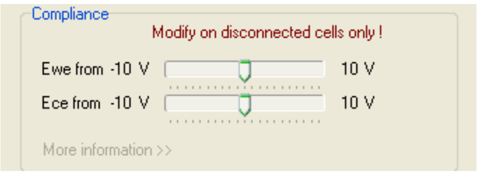
For this, used one of the techniques available in the « Ohmic Drop determination » folder.

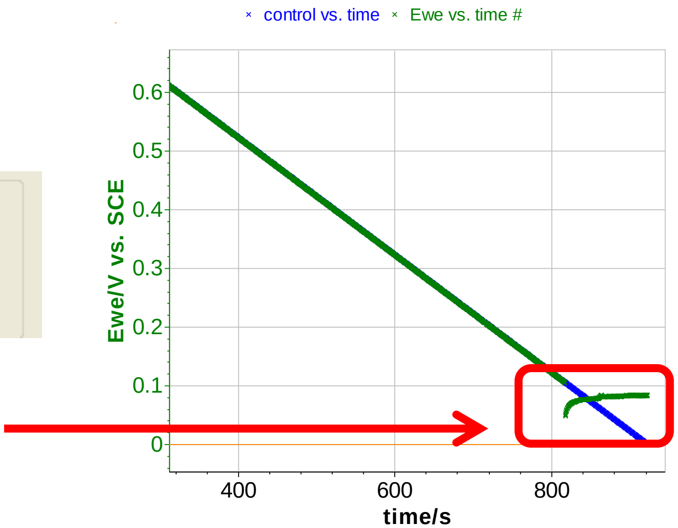


- **Troubleshooting.**

In some case, when the compliance is not wide enough .
Compliance needed can be checked by selecting Ece measurement in the « Cell characteristics ».
The compliance can be adjusted in the « advanced settings » only available for VMP3 family







It is easy to diagnose this kind of trouble by plotting the Econtrol (ramp of potential applied by the potential)and Ewe (measure dpotential) *vs* time. Ewe has to follow the control. If not, compliance or cell geometry has to be modified.