

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


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

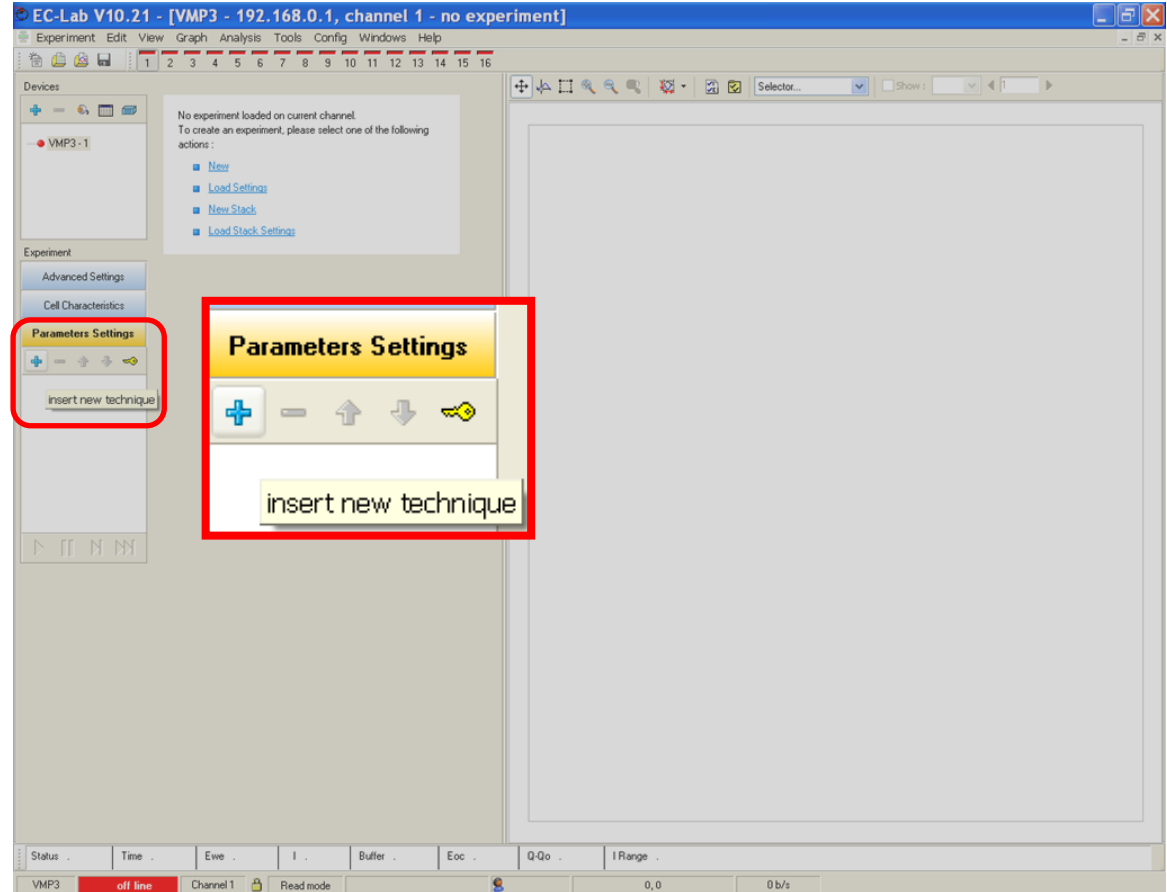
Only CV technique will be discussed hereafter but the information given in the presentation can be adapted to CVA, LP, MP techniques.

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:

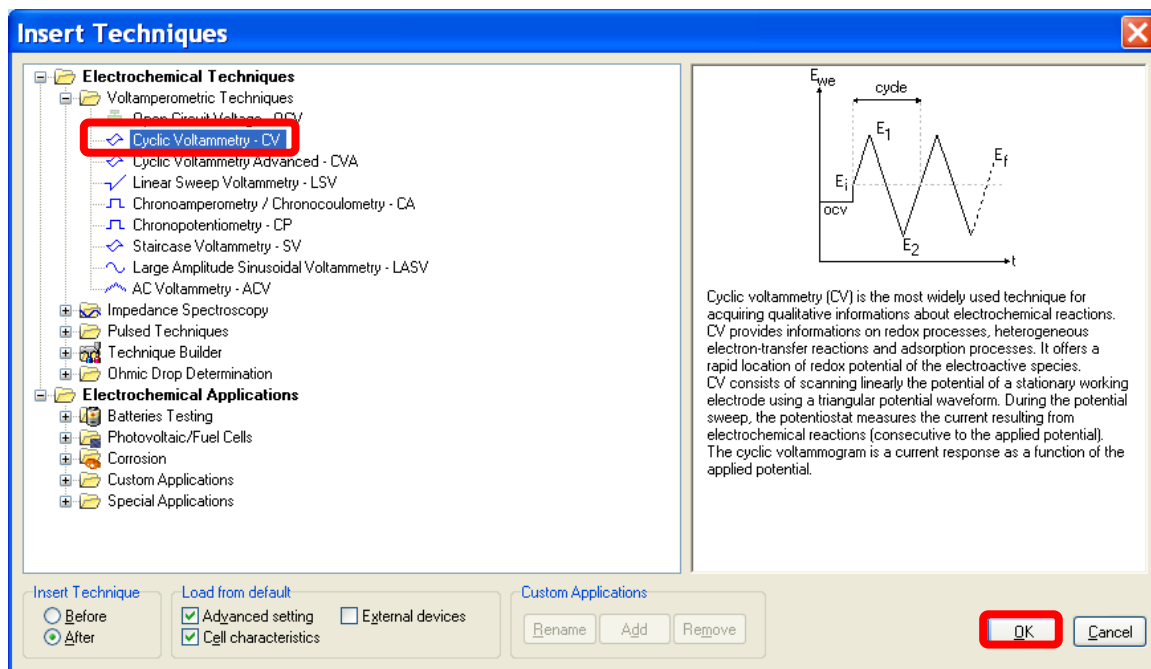
It is assumed that the computer and the instruments are connected. This is explained in the Getting Started named “EC-Lab®: Connection to the instrument(s) & Channel(s) selection”

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



Insert Techniques


- Electrochemical Techniques
 - Voltamperometric Techniques
 - Open Circuit Voltage - OCv
 - Cyclic Voltammetry - CV**
 - Cyclic Voltammetry Advanced - CVA
 - Linear Sweep Voltammetry - LSV
 - Chronoamperometry / Chronocoulometry - CA
 - Chronopotentiometry - CP
 - Staircase Voltammetry - SV
 - Large Amplitude Sinusoidal Voltammetry - LASV
 - AC Voltammetry - ACV
 - Impedance Spectroscopy
 - Pulsed Techniques
 - Technique Builder
 - Ohmic Drop Determination
- Electrochemical Applications
 - Batteries Testing
 - Photovoltaic/Fuel Cells
 - Corrosion
 - Custom Applications
 - Special Applications

Insert Technique: Before After





Load from default: Advanced setting External devices Cell characteristics

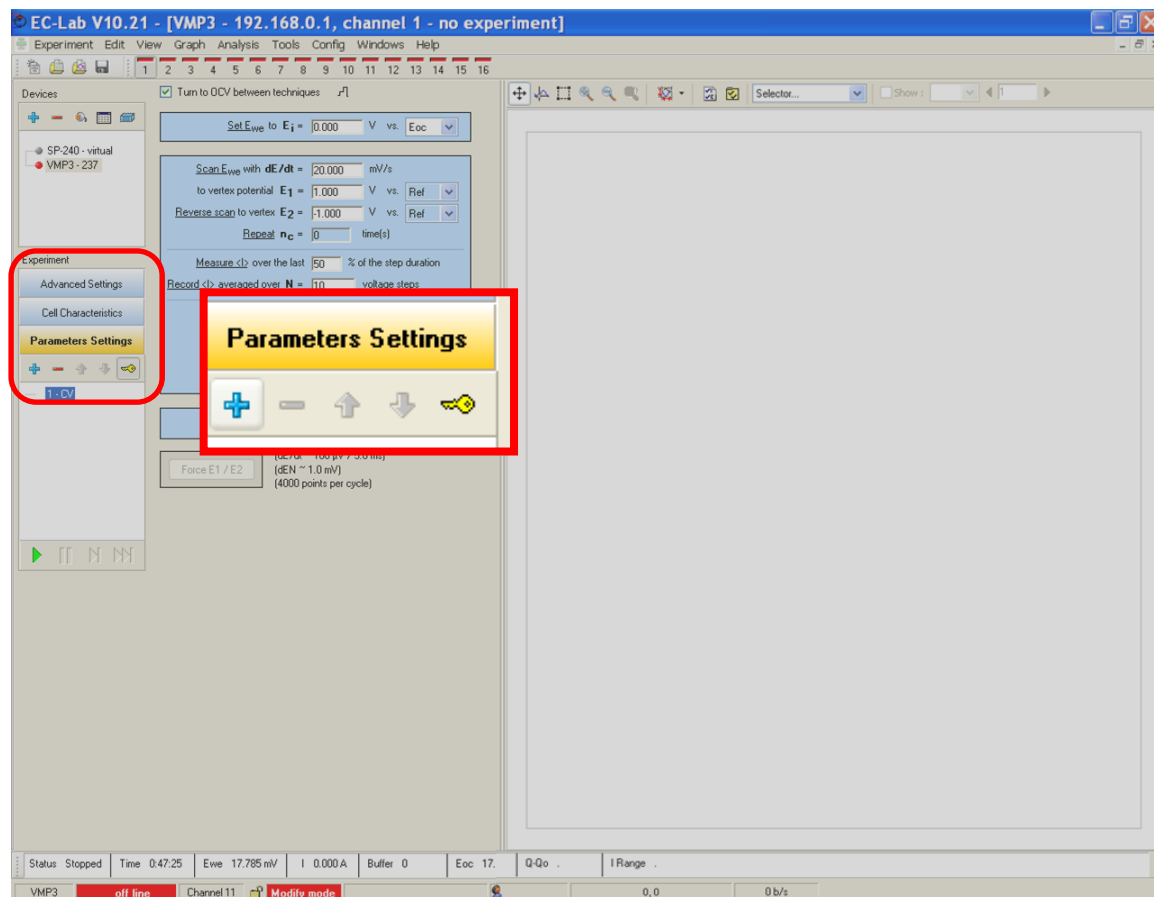
Custom Applications:

Cyclic voltammetry (CV) is the most widely used technique for acquiring qualitative informations about electrochemical reactions. CV provides informations on redox processes, heterogeneous electron-transfer reactions and adsorption processes. It offers a rapid location of redox potential of the electroactive species. CV consists of scanning linearly the potential of a stationary working electrode using a triangular potential waveform. During the potential sweep, the potentiostat measures the current resulting from electrochemical reactions (consecutive to the applied potential). The cyclic voltammogram is a current response as a function of the applied potential.

- 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



- 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

Compliance Modify on disconnected cells only !

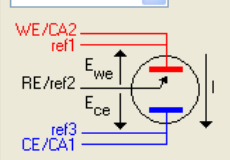
Ewe from -10 V 10 V
 Ece from -10 V 10 V

[More information >>](#)

Safety Limits

E_{we} max = V
 E_{we} min = V
 I_l = mA
 IQ-Qol = mA.h
 Analog IN 1 = V
 Analog IN 2 = V
 E stack slave max = V
 E stack slave min = V
 for t > ms

Electrodes Connection Modify on disconnected cells only !



Miscellaneous

Text export

Filter [Edit](#)

Smooth on points

Create one data file per loop (linked techniques only)

SP-300 family

Filtering

Ewe, I

Safety Limits

E_{we} max = V
 E_{we} min = V
 I_l = mA
 IQ-Qol = mA.h
 Analog IN 1 = V
 Analog IN 2 = V
 for t > ms

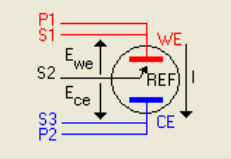
Channel

Floating
 Grounded

Ultra Low Current Option

High speed scan
Definition of high speed value depends on the current range used.

Electrodes Connection Modify on disconnected cells only !



Miscellaneous

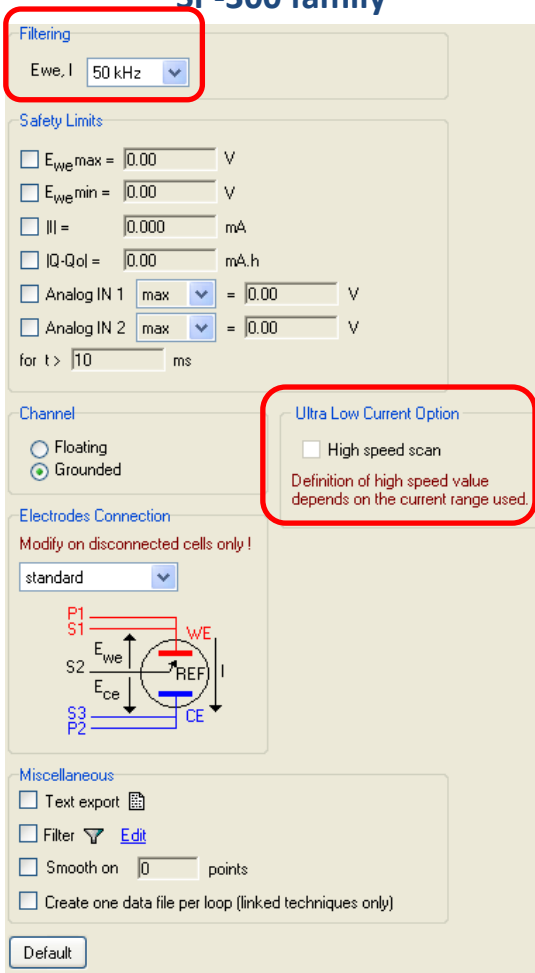
Text export

Filter [Edit](#)

Smooth on points

Create one data file per loop (linked techniques only)

SP-300 family



The screenshot shows the 'Advanced Setting' tab for the SP-300 family. The 'Filtering' section is highlighted with a red box, showing 'Ewe, I' set to '50 kHz'. The 'Safety Limits' section includes checkboxes for 'E_{We} max', 'E_{We} min', 'I_l', 'I_{Q-Qol}', 'Analog IN 1', and 'Analog IN 2', each with a numerical input field and a unit. The 'Channel' section has radio buttons for 'Floating' and 'Grounded'. The 'Ultra Low Current Option' section is also highlighted with a red box, containing a checkbox for 'High speed scan' and a note: 'Definition of high speed value depends on the current range used.' The 'Electrodes Connection' section has a dropdown menu set to 'standard' and a schematic diagram showing connections for P1, S1, WE, REF, I, S2, E_{We}, E_{ce}, S3, P2, and CE. The 'Miscellaneous' section includes checkboxes for 'Text export', 'Filter', 'Smooth on', and 'Create one data file per loop'. A 'Default' button is located at the bottom left.

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)

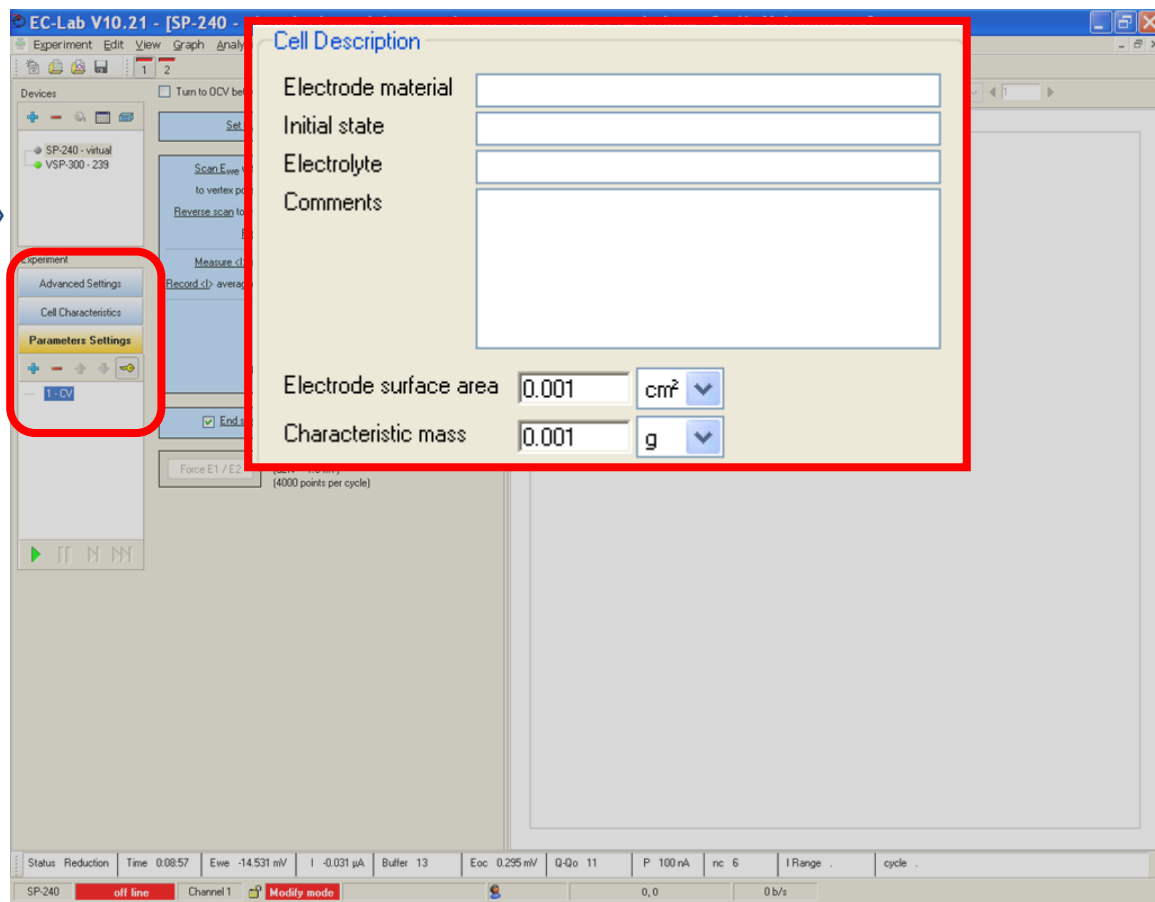
This option has to be ticked if the user works at very low level of current and at high scan rate.

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/cm²) instead of current (mA).



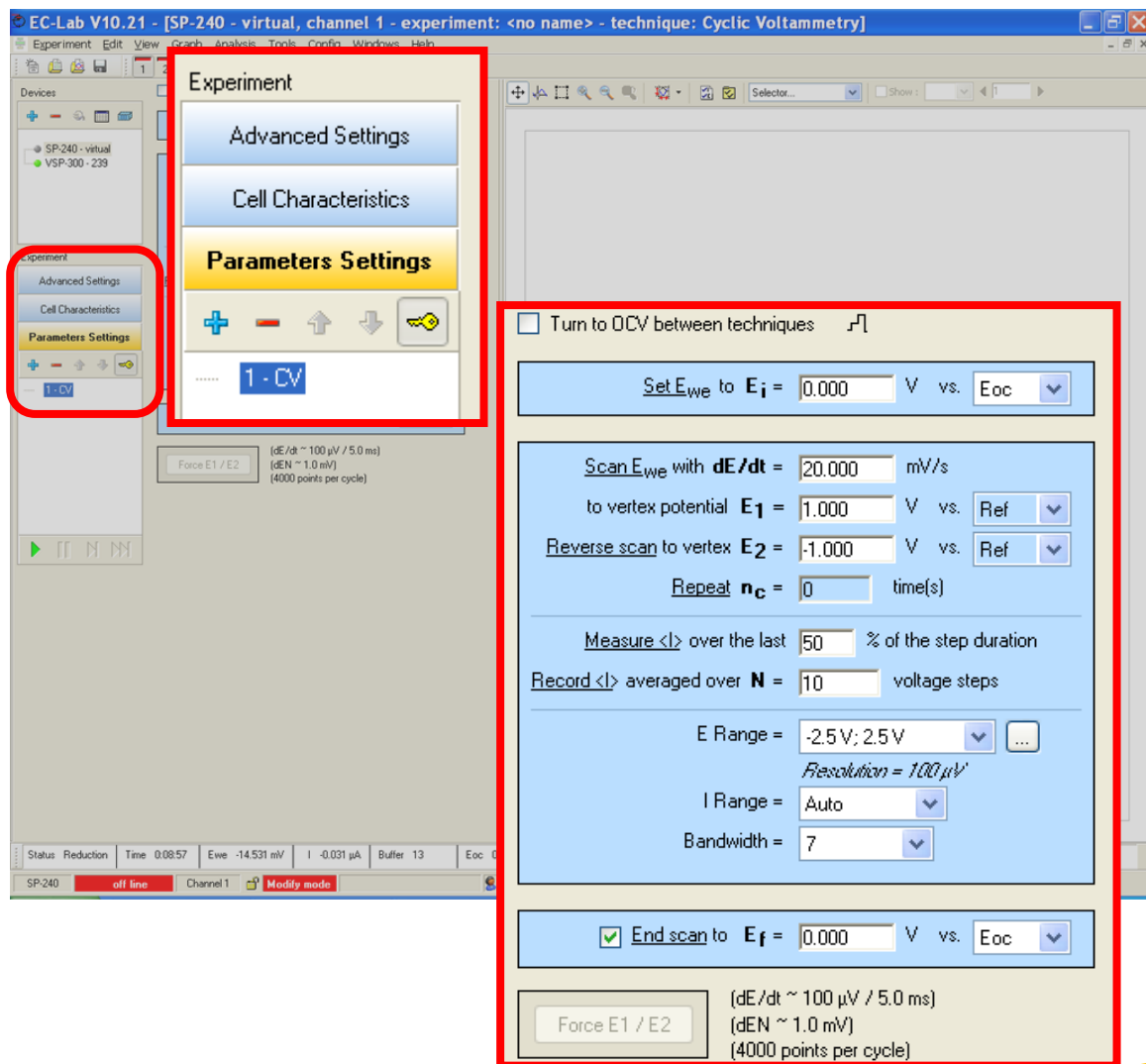
NOTE:
All these information are stored in the data file

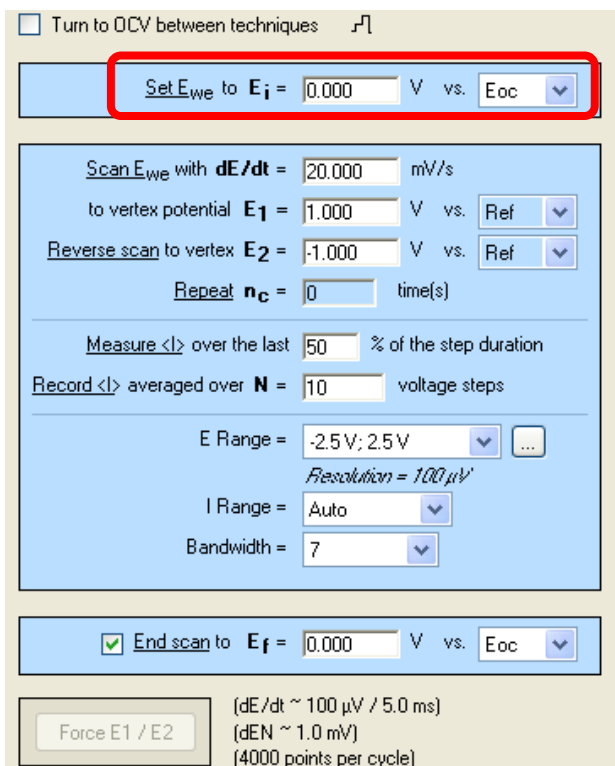
- Click on the **Parameters Settings** tab or directly on the technique CV in the list of technique. The technique is highlighted in blue.

- 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

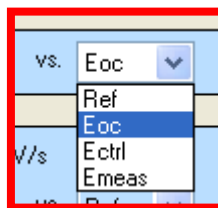
TIP: If the user would like to set a conditioning period before starting the voltage ramp, he has to set a CA technique before the CV or use the CVA technique.

NOTE: All the settings may be changed during the experiment (except I range, E range, bandwidth).





- 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:

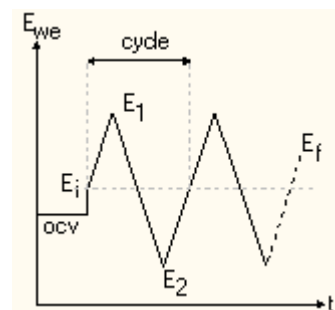
Set 0 V vs **Eoc** to avoid a current jump at the beginning of the CV.

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 E_i , E_1 , E_2 , E_f have to be in the E range. 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 E_{ref} , OCV may change whereas E_{ref} is absolute potential values .

Turn to OCV between techniques Γ

Set E_{we} to E_i = 0.000 V vs. Eoc \downarrow

Scan E_{we} with dE/dt = 20.000 mV/s
to vertex potential E_1 = 1.000 V vs. Ref \downarrow
Reverse scan to vertex E_2 = -1.000 V vs. Ref \downarrow
Repeat n_c = 0 time(s)

Measure $\langle I \rangle$ over the last 50 % of the step duration
Record $\langle I \rangle$ averaged over N = 10 voltage steps

E Range = -2.5 V; 2.5 V \downarrow 
Resolution = 100 μV

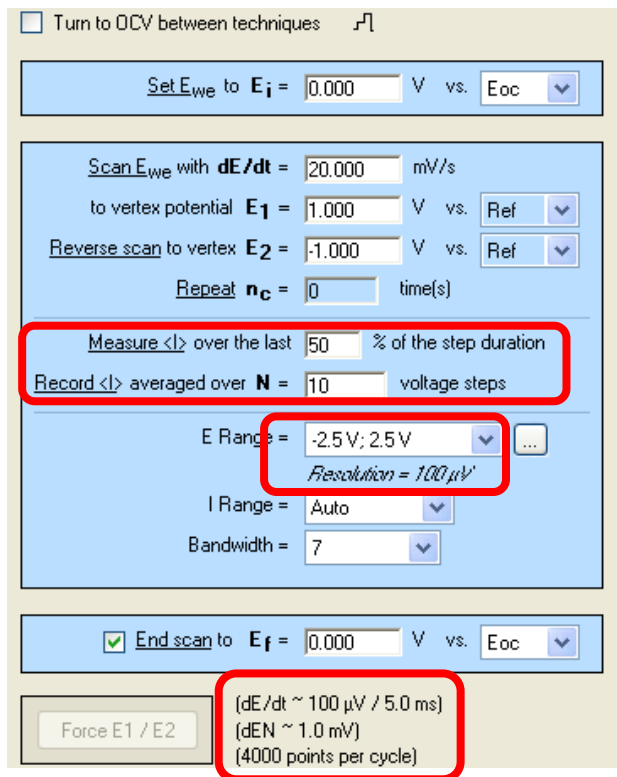
I Range = Auto \downarrow
Bandwidth = 7 \downarrow

End scan to E_f = 0.000 V vs. Eoc \downarrow

Force E_1 / E_2 (dE/dt ~ 100 μV / 5.0 ms)
(dEN ~ 1.0 mV)
(4000 points per cycle)

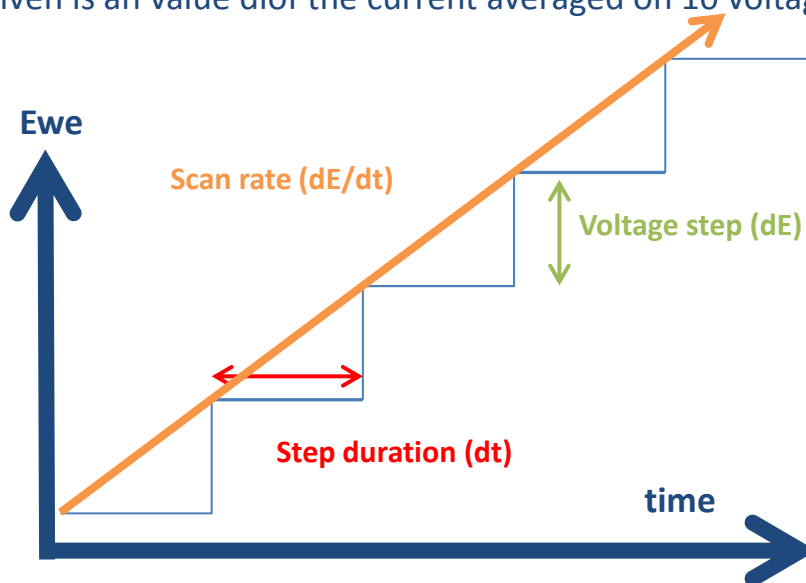
5- Set the CV technique

- 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.

Turn to OCV between techniques

Set E_{we} to E_i = 0.000 V vs. Eoc

Scan E_{we} with dE/dt = 20.000 mV/s
 to vertex potential E_1 = 1.000 V vs. Ref
 Reverse scan to vertex E_2 = -1.000 V vs. Ref
 Repeat n_c = 0 time(s)

Measure $\langle I \rangle$ over the last 50 % of the step duration
 Record $\langle I \rangle$ averaged over N = 10 voltage steps

E Range = -2.5 V; 2.5 V
 I Range = Auto
 Bandwidth = 7

End scan to E_f = 0.000 V vs. Eoc

Force E1 / E2 (dE/dt ~ 100 μ V / 5.0 ms)
 (dEN ~ 1.0 mV)
 (4000 points per cycle)

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.

Turn to OCV between techniques

Set E_{we} to E_i = 0.000 V vs. Eoc

Scan E_{we} with dE/dt = 20.000 mV/s
 to vertex potential E_1 = 1.000 V vs. Ref
 Reverse scan to vertex E_2 = -1.000 V vs. Ref
 Repeat n_c = 0 time(s)

Measure <I> over the last 50 % of the step duration
 Record <I> averaged over N = 10 voltage steps

E Range = -2.5V; 2.5V
Resolution = 100 μ V

I Range = Auto


Bandwidth = 7

End scan to E_f = 0.000 V vs. Eoc

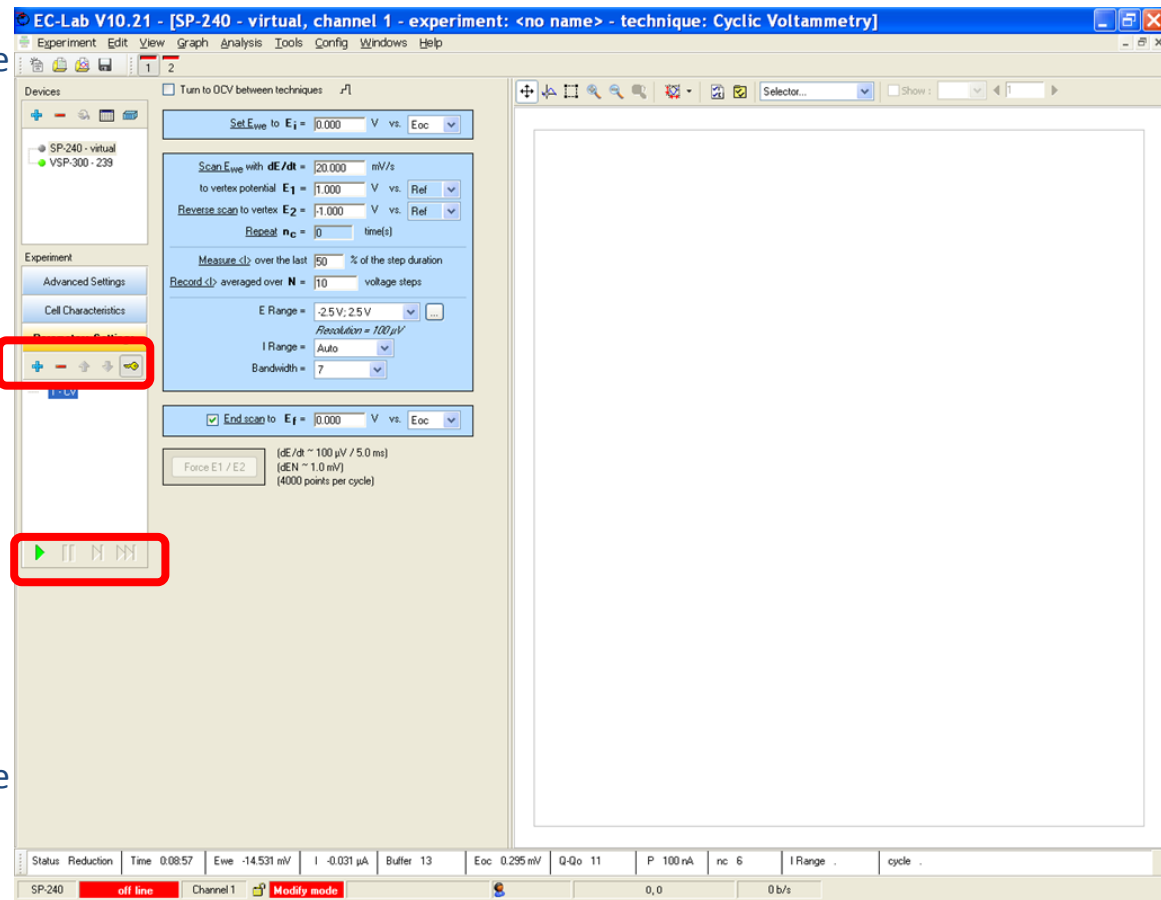
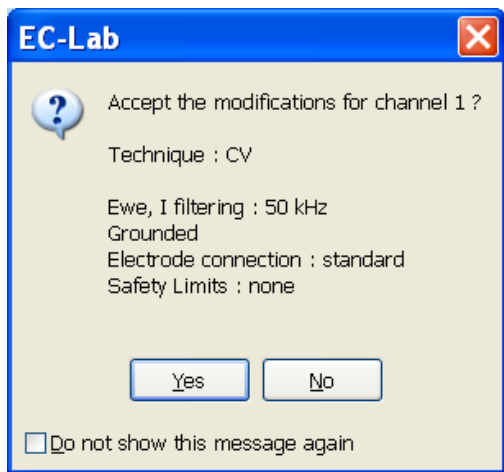
Force E1 / E2 (dE/dt ~ 100 μ V / 5.0 ms)
 (dEN ~ 1.0 mV)
 (4000 points per cycle)


- 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.

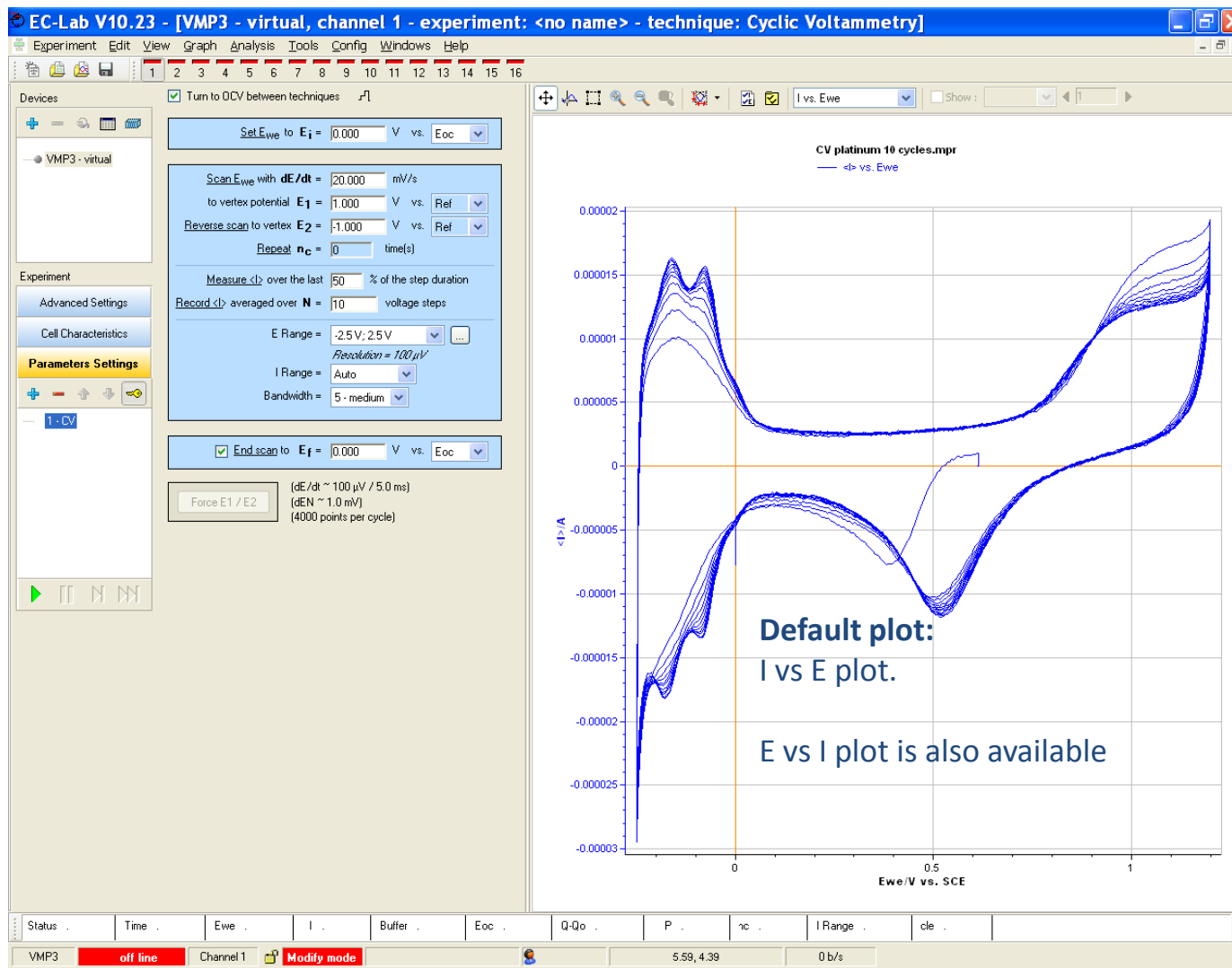
Click on  button to check if the settings are accepted

Some warning messages may come up.

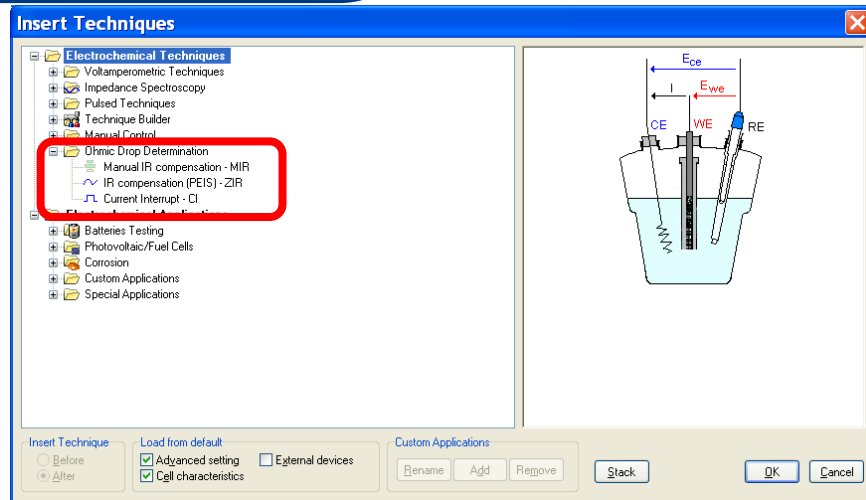


Click on the  button to start the experiment.

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

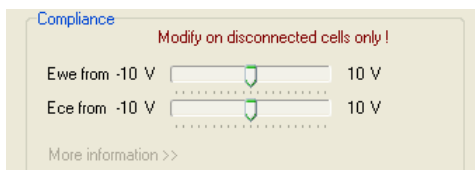
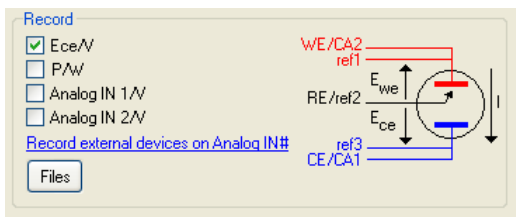


- **Ohmic drop** can be compensated.
For this, used one of the techniques available in the « Ohmic Drop determination » folder.



• 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.

