Application Note

Basics
AP-ORI03

Importance of Method Parameters in Electrochemical Tests

Properties

Display all | Details | Graph

Pot. Linear Voltammetry	
Potential 1 (mV)	-100, REF
Potential 2 (mV)	300, REF
Scan rate (mV/sec.)	0.45, 1, 0.45
Sampling rate	1:1
Maximum current (mA)	500
Minimum current (mA)	-500
Ohmic Drop Comp.	No
Maximum range	Auto
Minimum range	Auto
Open circuit at end	Yes
Save points	Yes
Analog Filter	Auto
Digital Filter	0
Auxiliary input	No

This Application Note describes about the importance of parameters of electrochemical methods on the results and curves.
At the end, the user will understand how to play with parameters in case of different applications and different samples.

Origalys Electrochem

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Introduction

Sometimes it happens that the users ask to have curves with less noise and better results. They see much noises or unusual results and curves that are not supposed to be. All the materials, connections, situations are checked and seems to be OK! So what is the problem?

In this application note we will see this can happen if the parameters of test is chosen wrong even if all the other situation seems ok. The parameters of Differential Pulse Voltammetry and Impedance methods are investigated, and the results are compared together. This application note could be generalized to other methods as well.

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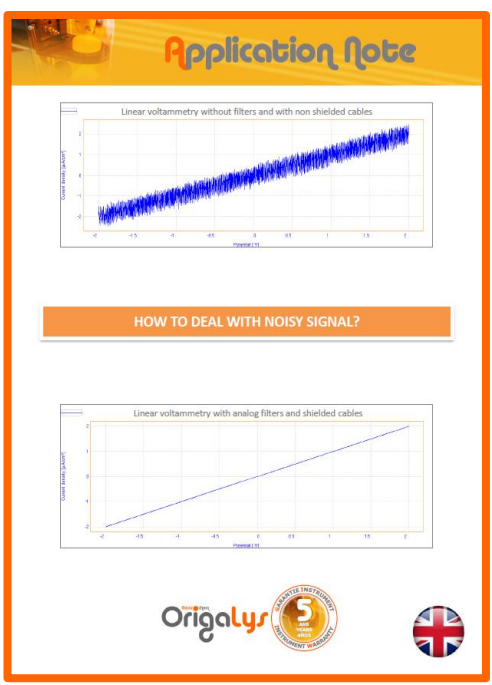


How to deal with noisy signal:

This document explains all the basics about noise in Electrochemical experiments.

Important parameters for all methods

There are common parameters in most of the OrigaMaster5 methods which play important role on result. Below you can find them with more description:

- **Maximum/Minimum current ranges:** Each sample during the tests (specially voltammetry methods) has the maximum and minimum current which shows in the I-V curve. It is very practical to know these limits and according to these limits define the maximum and minimum range in the method and change the default value.

> **TIPS:** We recommend:
> - to let the maximum range in AUTO. Indeed, it avoids to get any current overload.
> - And select a value for the minimum range

For example, in voltammogram figure 1, the maximum current is about 13 mA, so it is better to define minimum range = 100 mA (for OGF01A or OGS100), 50 mA (for OGF500) or 20 mA (for OGS200).

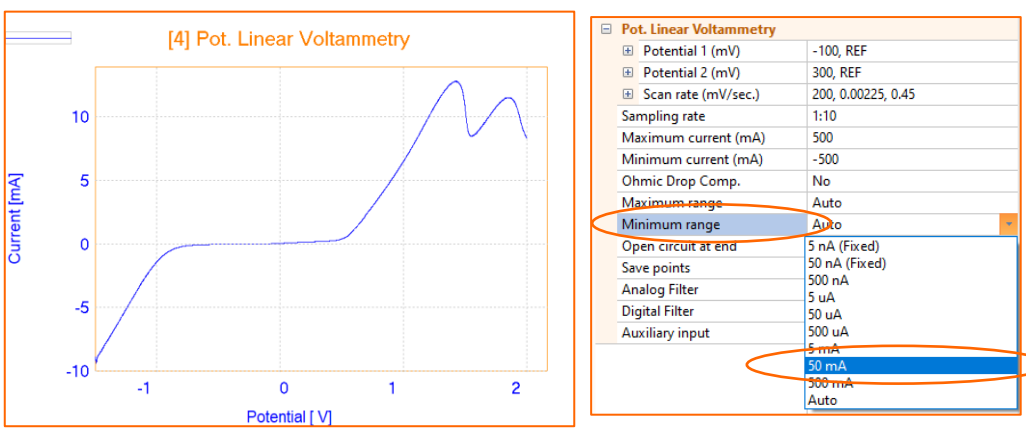


Figure 1: Linear Voltammetry with optimized current range (OGF500)

- **Sampling Rate:** The default parameter of software is 1:1 which means all the points will be saved and shown. But in long term tests with too many points, this parameter can be changed and be lower. For example 1:5, which means the software will divide the number of points by 5. This is very practical in case of facing "memory full" error.

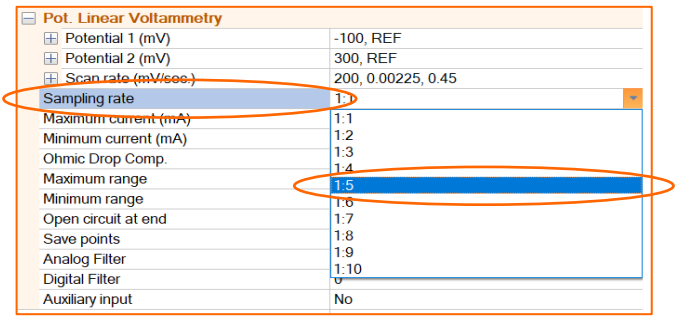
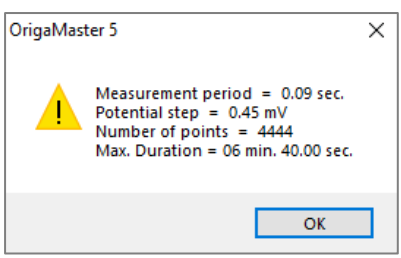


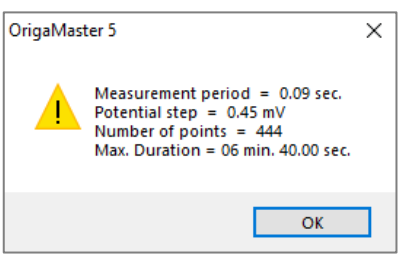
Figure 2: Sampling rate is an important parameter for long term tests

Example: Linear Voltammetry

Scan rate = 5 mV/s
Duration step = 0.09 s
Potential step = 0.45 mV
Sampling rate = 1:1
Number of points
4,444



Scan rate = 5 mV/s
Duration step = 0.09 s
Potential step = 0.45 mV
Sampling rate = 1:10
Number of points
444



- **Analog Filters** are used extensively in sensor signal processing. The main use of these signal processing techniques is reduced the noise value.

    - It is set as Auto by default. Sets the time constant value of the measurement system.
    - 1 µsec. is the lowest value whereas 1 sec. is the biggest value.

In AUTO, the value of the Analog filter is always related to the measurement period (Step duration parameter in the scan rate). In the below example:

- Step duration = 0.00225 sec. = 2.25 msec.
- As step duration > 1 msec., then filter = 100 µsec.

TIPS: For the low scan rate, it is recommanded to select a filter of 1 msec. or 10 msec.

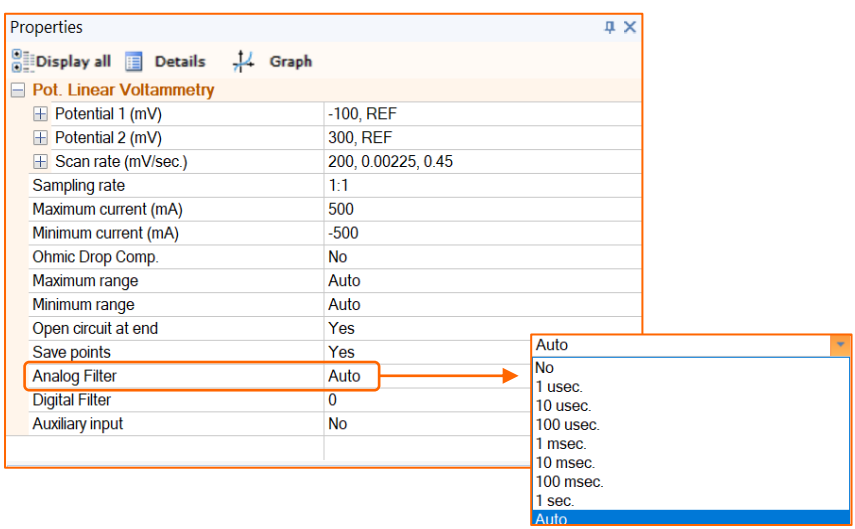


Figure 3: Analog Filters, useful tools for redusing noises

- **Digital Filters** are used extensively in sensor signal processing. The main use of these signal processing techniques is reduced the noise value.

    - This parameter reduces the noise amount through performing average during live test like as smoothing. The value could be defined from 1 to 20. The maximum filtering will be performed if the parameter defined as 20.

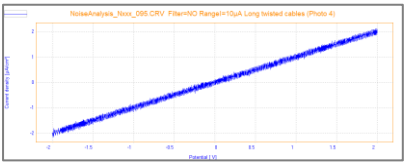


Figure 4: Digital Filter = 0

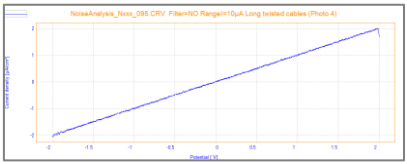


Figure 5: Digital Filter = 5

TIPS: After the experiment, in the Curve tab, General, it is possible to put add smoothing in X, Y1 and Y2 (from 0 to 20).

So, minimum Digital filter is 0 and maximum is 40

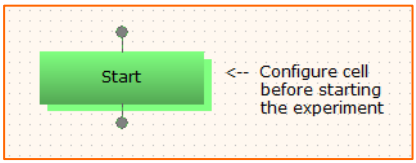
Properties

Display all | Details | Graph

Pot. Linear Voltammetry	
Potential 1 (mV)	-100, REF
Potential 2 (mV)	300, REF
Scan rate (mV/sec.)	200, 0.00225, 0.45
Sampling rate	1:1
Maximum current (mA)	500
Minimum current (mA)	-500
Ohmic Drop Comp.	No
Maximum range	Auto
Minimum range	Auto
Open circuit at end	Yes
Save points	Yes
Analog Filter	Auto
Digital Filter	0
Auxiliary input	No

Figure 6: Digital Filters, useful tools for redusing noises

Important parameters in the START



- **Delay Before Disjunction:** Normally, if the current measured becomes higher than the maximum range selected, the electrodes are disconnected and the sequence in progress is stopped (disjunction of the system). Nevertheless, you can maintain the electrodes connected for a certain time (Delay before disjunction) if such a current overshoot occurs.
- By introducing a delay before disjunction, you prevent the sequence from being stopped after a sudden change in current especially when working in the low current range.

TIPS: The Delay before disjunction parameter is not significant when working at low scanning rates (corrosion studies for example).

Properties

Display all | Details | Graph

Initialization - Cell configuration	
Electrodes connected with	OGS/OGF/LDS
Connection cell on	3 electrodes
Temperature sensor	No
Settings Instruments	
Delay before disjunction (msec.)	20
Auto ranging delay (msec.)	200
Bandwidth limit	No
Stopping criteria	
Use the potential limits	
Maximum potential (mV)	15000
Minimum potential (mV)	-15000
Use the current limits	
Maximum current (mA)	100
Minimum current (mA)	-100
Use external control	

Figure 7: Delay before disjunction in START

- **Auto Ranging Delay:** Setting the time of automatic change of current range. This delay is only active from an upper current range to a lower current range.
- For example when switching from the 1mA range to 10mA, the timer is started and enabled, then no turning back to the range 1mA is authorized until the end of the duration of the delay.
- This functionality avoids continuous range changes when the current measurements are very noisy, especially when working in low current range.

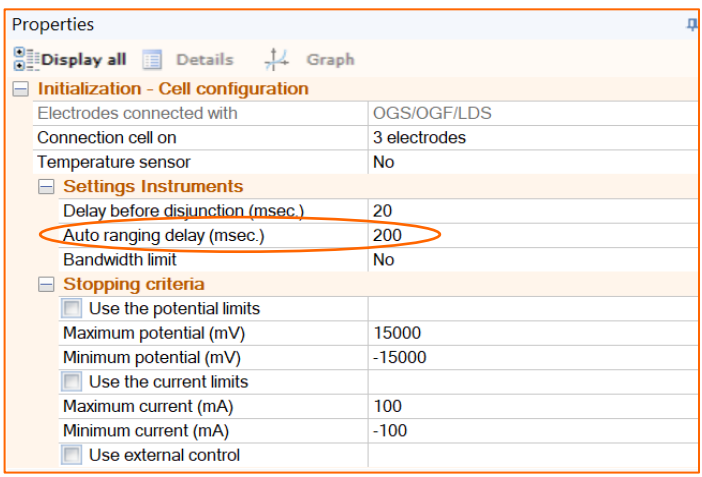


Figure 8: Auto ranging delay in START

NOTE: In following pages more details about two practical methods and the important parameters are investigated. It could be understood at the end that how much the parameters and their values play significant role in results.

Differential Pulse Voltammetry (DPV)

One of the quantitative methods for analysing electrochemical reactants. Its detection limit is 10-8 M. Definition of correct parameters directly affect the result while measuring very low concentrations. Important default parameters:

Properties

Display all | Details | Graph

Pot. Differential pulse	
Potential 1 (mV)	-100, REF
Potential 2 (mV)	300, REF
Scan rate (mV/sec.)	25
Step (mV)	1
Pulse (mV)	60
Pulse time (msec)	20
Minimum range	50 uA
Maximum range	5 mA
Digital Filter	0
Open circuit at end	Yes
Save raw data	Yes

Figure 9: Default parameters of Differential Pulse Voltammetry

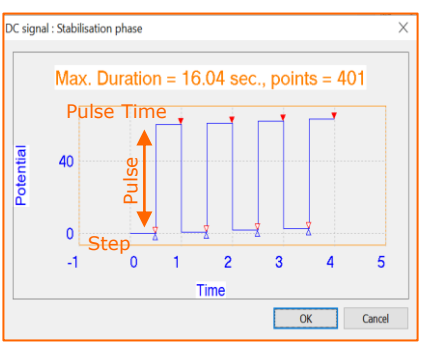


Figure 10: Schema of the applied potential during DPV method

Step Potential, **Pulse** and **Pulse Time:**

The higher the value of these parameters, the sharper the current of the peak will be. But it is needed to fined the optimised value of these parameters according to sample and application.

For example, in polarography methods which are used for trace analysis of heavy metals, the pulse value could be more than 120 mV as well as the pulse time which could be even more than 100 msec.

NOTE: So according to sample and application, all these parameters could be changed, and it is not advised to always perform electrochemical tests based on default parameters.

Pot. Dynamic EIS (Impedance)

The default parameters of Impedance method are as below:

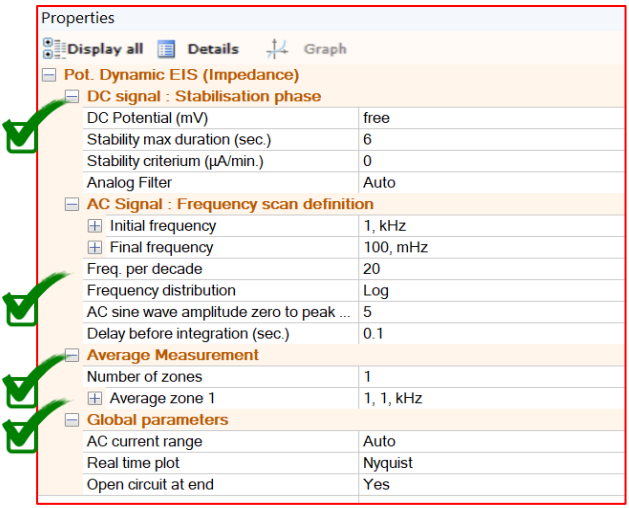


Figure 11: Parameters of Impedance method

- **Stability Duration** is directly influence on reducing the scattering in Nyquist results, specially in corrosion analysis. This helps user to have much stable working electrode. The higher the stabilization time, the better the Nyquist curve.
- **AC Sine Wave** is the amplitude of AC potential wave. The higher the amplitude, the lower noise will be seen, but there is always an optimized value for each sample and application.
- **AC Current Range** is the very important parameter which all users need to consider. It is highly recommended to use fixed current range which will reduce the scattering in Nyquist plots. Through performing CV before EIS test, the Maximum Range could be extracted.
- **Average** performs average of the measured values. For example, if it is defined as 5, it means it will perform average of 5 points of measurements and then show the result on the curve.

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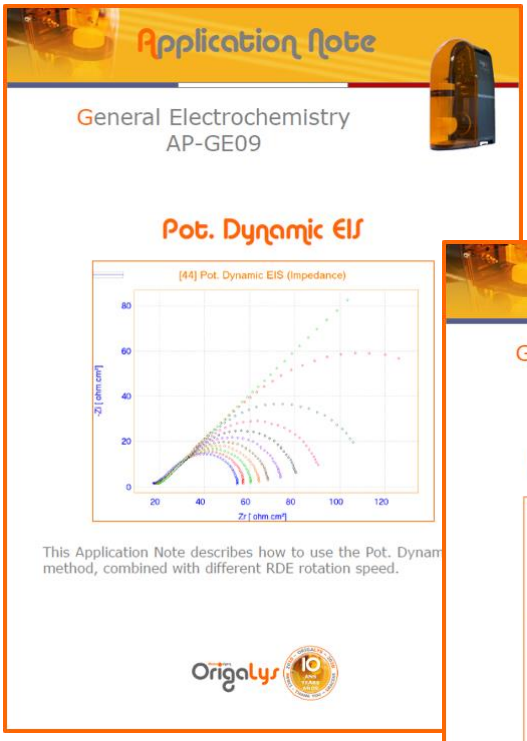

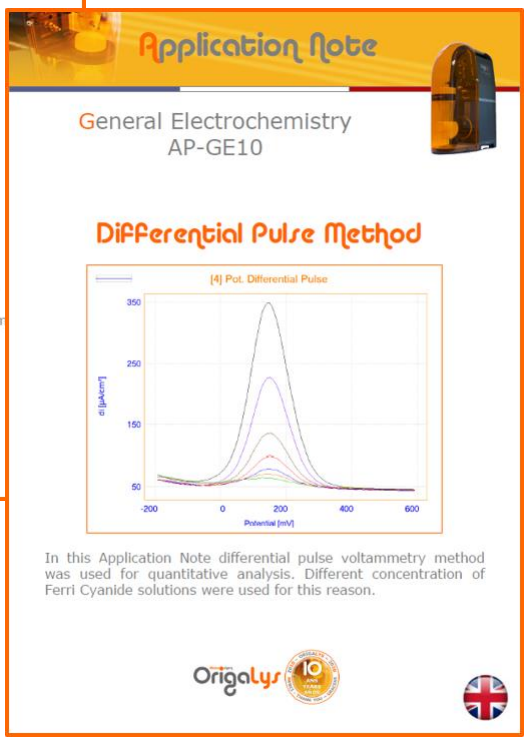

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