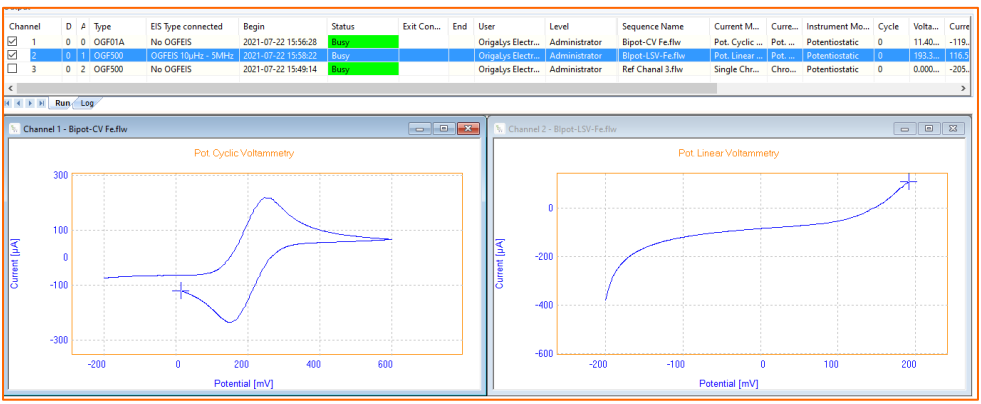
Application Note

General Electrochemistry
AP-GE14

Various electrochemical methods of study on two working electrodes with Bipotentiostat configuration



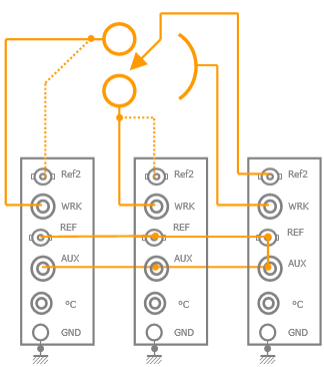
In this application note bipotentiostat configuration was used to perform different electrochemical tests like cyclic voltammetry, Linear voltammetry or pitting corrosion on different samples using two working electrodes. At the end, user will understand how to configure bipotentiostat tests on 2 working electrodes in parallel with OrigaFlex potentiostats.

INTRODUCTION

In the normal 3-electrodes set-up, one working electrode is used for electrochemical analysis. But in a bipotentiostat, two working electrodes will be used and controlled separately. In that case, one counter and one reference electrode will be immersed in solution, then potential or current is imposed separately on WRK1 and WRK2.

Bipotentiostats provide second working electrode which can be used for any electrochemical investigation. For example it could be possible to perform CV on WRK1 and DPV on WRK2.

In order to have bipotentiostat configuration, three OrigaFlex modules must be connected to a drive unite as it could be seen in figure 1.
Reference and Auxiliary electrodes are connected to channel 3, then Working electrode 1 is connected to channel 1 and working electrode 2 is connected to channel 2. The cables connected in green sockets (figure 1b) are necessary to have configuration shown in figure 1a.



(a)

(b)

Figure 1: scheme (a) and real (b) configuration of bipotentiostat

TIPS: If user does not have the connectors as shown in figure 1b, it is possible to connect bipotentiostat configuration by ordinary cables. For more information, please contact us.

1 Bipotentiostat Test on Dummy Cell

In the first section, bipotentiostat tests were run on dummy cell. In next section more tests were run on real samples.

After connecting the 3 x OrigaFlex as Bipotentiostat mode (like figure 1), the cables are connected from instruments to dummy cell as shown in figure 2.

Figure 2: Connecting Dummy Cell to Bipotentiostat

As it is sown in figure 1a, the REF2 and WRK of channel 3 are respectively connected to Reference and Auxiliary sockets of dummy cell. About WRK1 and WRK2 it must be noticed that which test is going to be performed. For example, in figure 2 WRK1 is connected to CV socket and WRK2 is connected to LIN socket of dummy cell enabling user to perform cyclic voltammetry on channel 1 and linear voltammetry on channel 2.

General Parameters (for channel 3)

In all Origalys Bipotentiostat experiments, the role of channel 3 is always to impose a virtual ground potential on the system's reference electrode. This is why a potential of 0-mV is imposed on this channel by the "**Chrono Amperometry Expert**" method for a long period (10 hours for example).

Figure 3 shows the related parameters.

A non-zero potential can be imposed by the 3rd channel, this will shift the potential of the other channels (for more information, please contact us).

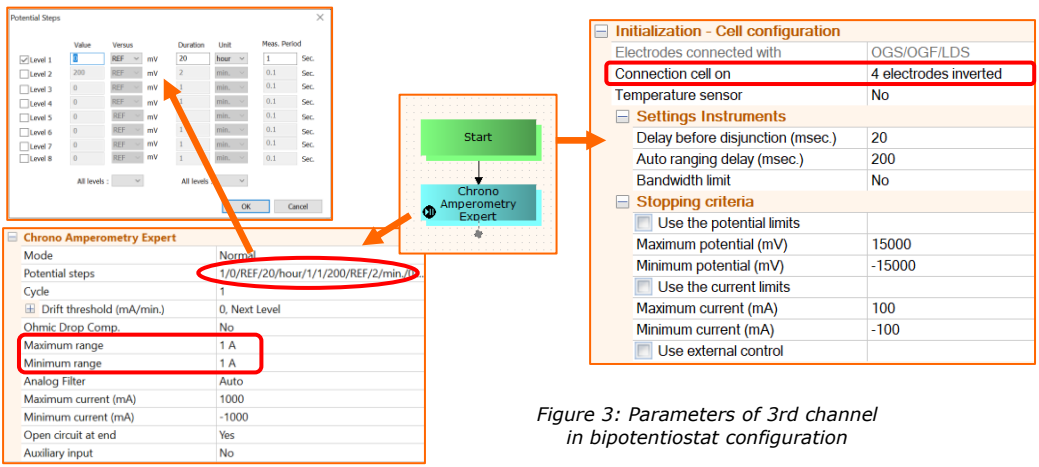


Figure 3: Parameters of 3rd channel in bipotentiostat configuration

Important NOTES:

1- For the 3rd channel the electrode configuration must be defined as **"4 electrodes inverted".**

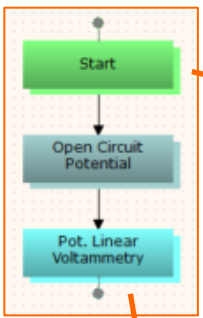
2- **The 3rd channel must be started before** running any experiment on channel 1 or 2.

3- The duration of chrono amperometry on 3rd channel **must be long enough (here 20 h)** to cover the whole duration of measurements on other channels.

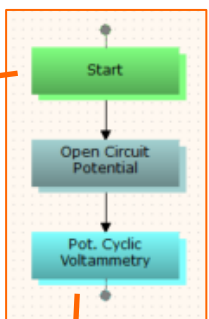
4- **The current range must be in fixed mode, at its maximum value** (in this example 1A for an OGF01A), in order to avoid a possible risk of "Potential overload" error.

Parameters of CV and LSV methods on dummy cell

After definition of parameters of chrono amperometry for channel 3, then Cyclic and Linear Sweep Voltammetry were performed on channel 1 and 2 respectively. Figure 4 shows the parameters of these two mentioned methods on bipotentiostat mode. There are both performed on 3 electrodes mode.



Initialization - Cell configuration	
Electrodes connected with	OGS/OGF/LDS
Connection cell on	3 electrodes
Temperature sensor	No
Settings Instruments	
Delay before disjunction (ms...	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	
Variables initialization	



(a)- channel 1

(b)- channel 2

Pot. Cyclic Voltammetry	
Potential 0 (mV)	-2500
Potential 1 (mV)	3500
Potential 2 (mV)	-2500
Scan rate (mV/sec.)	100, 0.018, 1.8
Sampling rate	1:1
Maximum current (mA)	1000
Minimum current (mA)	-1000
Ohmic Drop Comp.	No
Maximum range	Auto
Minimum range	Auto
Analog Filter	Auto
Digital Filter	0
Cycle	3
Open circuit at end	Yes
Save points	Yes
Auxiliary input	No

Pot. Linear Voltammetry	
Potential 1 (mV)	-2500, REF
Potential 2 (mV)	3500, REF
Scan rate (mV/sec.)	100, 0.0135, 1.35
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 4: Parameters of cyclic and Linear Voltammetry tests on bipotentiostat mode

Results and discussion

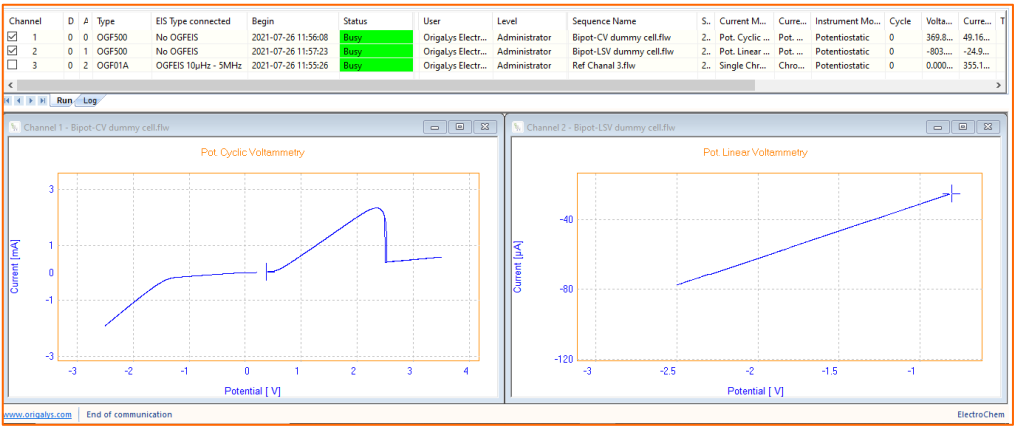


Figure 5: Online curves of bipotentiostat test on dummy cell

Figure 5 shows online curves of CV (related to channel 1) and LSV (related to channel 2).

After finishing the performance, all the results could be saved, opened and analyzed in both OV2 and OM5 software.

NOTES:

Electrode configuration for channel 1 and 2 could be defined as 3 or 4 electrodes Normal. It depends on sample and the method of analyze.

2 Bipotentiostat Test on real sample

In this part, bipotentiostat tests were performed on real well known redox couple; Ferri/Ferro Cyanide 0.5 M. On the first channel, cyclic voltammetry test were run, whereas Linear voltammetry test were run on second channel.

Like previous section, on the channel 3, single chrono amperometry was performed by imposing 0 mV as potential (figure 3).

Figure 6 shows how electrodes WRK1, WRK2, REF and AUX were connected from potentiostats to electrodes.

Figure 6: Bipotentiostat manipulation on Ferri/Ferro Cyanide couple

NOTES:

Current on WRK1 + Current on WRK2 < Current on AUX.

The current of 3rd channel is summation of channel 1 and channel 2.

Parameters of CV, LSV and DPV methods on Ferri/Ferro Cyanide

Different tests of voltammetry like: CV, LSV and DPV were run on Ferri/Ferro Cyanide solution. Figure 7 shows the flow chart related to bipotentiostat tests for channel 1 and 2. The parameters of "Start" box are like figure 4 because here the 3 electrodes configuration were applied as well.

Initialization - Cell configuration	
Electrodes connected with	OGS/OGF/LDS
Connection cell on	3 electrodes
Temperature sensor	No
Settings Instruments	
Delay before disjunction (msec.)	200
Auto ranging delay (msec.)	20000
Bandwidth limit	No
Stopping criteria	
Variables initialization	

(a) « Start » box

Pot. Differential Pulse	
Potential 1 (mV)	-200, REF
Potential 2 (mV)	600, REF
Scan rate (mV/sec.)	25
Pulse (mV)	90
Pulse time (msec)	20
Minimum range	500 nA
Maximum range	5 mA
Digital Filter	3
Open circuit at end	Yes
Save raw data	Yes

(b) DPV parameters

Pot. Cyclic Voltammetry	
Potential 0 (mV)	-200
Potential 1 (mV)	600
Potential 2 (mV)	-200
Scan rate (mV/sec.)	20, 0.0225, 0.45
Sampling rate	1:2
Maximum current (mA)	500
Minimum current (mA)	-500
Ohmic Drop Comp.	No
Maximum range	Auto
Minimum range	Auto
Analog Filter	Auto
Digital Filter	2
Cycle	3
Open circuit at end	Yes
Save points	Yes
Auxiliary input	No

(c) CV parameters

Pot. Linear Voltammetry	
Potential 1 (mV)	-200, REF
Potential 2 (mV)	600, REF
Scan rate (mV/sec.)	20, 0.0225, 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	2
Auxiliary input	No

(d) LSV parameters

Figure 7: Parameters of Voltammetric tests on Ferri/Ferro Cyanide couple by Bipotentiostat mode

Results and discussion

Figure 8: Online curves of bipotentiostat tests on Ferri/Ferro Cyanide
1: (a) channel 1 is for CV and (b) channel 2 is for DPV
2: (a) channel 1 is for CV and (b) channel 2 is for LSV

Figure 8 shows online curves of Cyclic voltammetry, Differential Pulse and Linear Sweap Voltammetry on Ferri/Ferro cyanide on two working electrodes.

Application on Corrosion study

One of the most popular application of bipotentiosat configuration is corrosion study. Thanks to this, it is possible to study two different alloys of metals in the same solution (for example salty water, sea water or solutions containing corrosion inhibitors) and analysing the corrosion behaviour of these alloys at the same time.

Figure 9 corresponds performing pitting corrosion on different alloys in NaCl solution 30 gr/L: Stainless Steel 316L as WRK1 (channel 1) and Carbon Steel XC38 as WRK2 (channel 2). Parameters of tests for both channels were the same (figure 10).

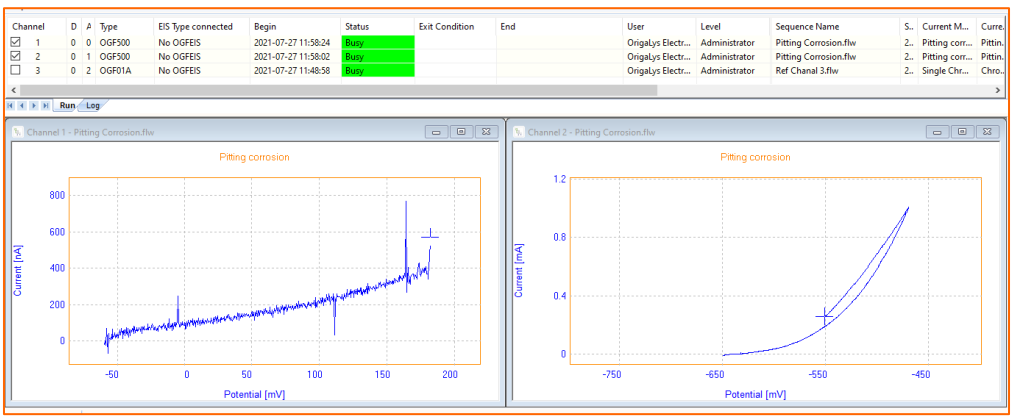
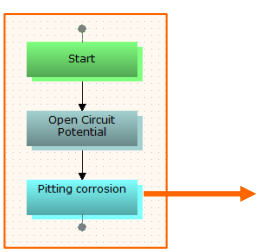


Figure 9: Online curves of bipotentiostat test on metal alloys for corrosion study



Pitting corrosion	
Initial potential (mV)	-25, FREE
Scan rate (mV/sec.)	1, 0.45, 0.45
Current threshold (mA)	1
Potential threshold (mV)	1000, REF
Hold potential duration (sec.)	2
Reverse the scan direction	Yes
Stop reverse potential (mV)	-25, FREE
Stop reverse current (mA)	-2
Ohmic Drop Comp.	No
Maximum range	Auto
Minimum range	Auto
Digital Filter	0
Analog Filter	Auto
Open circuit at end	Yes

Figure 10: Parameters of Pitting Corrosion test on two alloys (Stainless Steel 316L and Carbon Steel XC38)

INSTRUMENT AND ELECTRODES

Figure 11: Bipotentiostat configuration

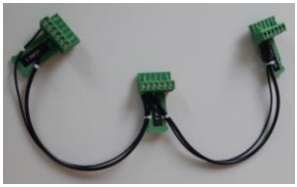


Figure 12: Cables and connections regarding bipotentiostat configuration

Figure 13: OrigaTest-Dummy Cell

Electrode setup	
Reference Electrode (REF)	Calomel Type: OGR003
Work Electrode (WRK)	- 2 x Pt tip 3 mm - Stainless Steel 316L tip 5 mm (EMEDT316LD5) - Carbon Steel XC38 tip 5 mm (EMOGTXC38D5CIAL)
Counter Electrode (AUX)	Platinum Wire Type: OGV005
Electrolyte	- KCl 10 gr/L - NaCl 30 gr/L - Ferri/Ferro Cyanide 0.05M
Instrument	- Drive Unit - OGF500 - OGF500 - OGF01A
Software	OrigaViewer

REF
Calomel

AUX
Platinum wire

WRK
Pt tip
Stainless Steel 316L tip
XC38 tip

OrigaLys ElectroChem SAS
Les Verchères 2
62A, avenue de l'Europe
69140 RILLIEUX-la-PAPE
FRANCE
☎ **+33 (0)9 54 17 56 03**
+33 (0)9 59 17 56 03
contact@origalys.com