# Series EIS Test by Cyclic Voltage step using sequence file

### Purpose

2 mM potassium ferricyanide(K3Fe(CN)6) with 1 M potassium nitrate(KCN) in water is used for typical cyclic voltammetry test. The voltage scanning range is 0V to 600mV. EIS test will be done by each 100mV step after constant voltage is applied and reverse scan. With this example, you can test your electrochemical cell by same manner to get EIS graph per specific voltage automatically. This application note guides to make this experiment using sequence file to demonstrate how to use sequence file.

This demonstration's test condition is;

- Start potential: 600mV
- Middle potential: 0V
- Final potential: 600mV
- Voltage step: 100mV
- EIS measurement at each potential step
  - Initial frequency: 100kHz
  - Final frequency: 0.1Hz
  - Amplitude: 10mV
  - Points per decade: 10

### Preparation

- ZIVE SP/MP electrochemical workstation
- Pt working electrode
- Ag/AgCl reference electrode
- Graphite rod counter electrode
- Universal Electrode Holder
- Cell vial
- Electrolyte:2 mM potassium ferricyanide(K3Fe(CN)6) with 1 M potassium nitrate(KCN) in water.

#### Preparation of the Test Solution

- 1. Weigh 16.5 mg potassium ferricyanide and place in a 25 mL volumetric flask.
- 2. Weigh 2.53 g potassium nitrate and add to the same volumetric flask.
- 3. Add about 20 mL deionized water to dissolve the potassium ferricyanide and potassium nitrate.
- 4. Dilute to 25 mL with deionized water.

## **Cell Connection**

Platinum Working Electrode (Green lead & Blue lead) Graphite Counter Electrode (Red lead) Ag/AgCl Reference Electrode (White lead)

Add 10-15 mL of the ferricyanide solution to the cell vial and place in the cell holder. Insert Pt working electrode on one of hole in cell top and screw right the electrode holding screw. Insert Graphite counter electrode as same way of Pt electrode Insert Ag/AgCI reference electrode as same way of Pt electrode (Reference electrode body is fragile. Be carefu

Insert Ag/AgCl reference electrode as same way of Pt electrode (Reference electrode body is fragile. Be careful on holding screw. Too much forced screwing might break the electrode)



photo: Universal holder (model: UEH)

#### Procedure

- 1. Turn the Power switch on the ZIVE SP/MP electrochemical workstation
- 2. Open the SM software by clicking the SM icon. The following progress box will appear, and will show the progress of checking instrument configuration and communication between ZIVE SP/MP electrochemical workstation and PC.



If the link is successfully connected, Click "OK" button on the box then the progress box will automatically disappear and SM software will appear. If the link failed, The following progress box will display then click the "Retry" button.



If the link failed again after clicking "Retry" button, you need to check USB cable connection.



3. Click New Sequence function icon (or select Sequence function-New on Experiment menu)

#### Then you can see the following menu

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4. Click Add button Add

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The above 3 step is for constant voltage applying and EIS measurement. 3<sup>rd</sup> step is to mark cycle. This cycle mark will be used to separate each EIS data when you analyze EIS data using Zman software. If you do not insert Loop(cycle) step after EIS measurement, You must separate each EIS data by manually.

To copy the above 3 steps, drag mouse from step numbers them selected region will be changed with yellow color

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Change the voltage value from 600mV to 500mV at step4.

Copy step1 to step 6 and paste it into step 7 to step12 and change voltage value as 600mV,500mV,400mV,300mV,200mV,100mV,00mV,100mV,200mV,400mV,500mV,600mV as the follows.

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- 5. Click "Save" button to save the technique file which contains the above parameter and setting file name.
- 6. You can assign this technique file to channel(s) which you want by click "APPLY" button or select schedule file using file selection on "Single channel control/monitor" or "multi channel control/monitor"

If you click "apply" button on technique menu, You can see the following channel selection menu.



Select channel and click OK then this technique file will be assigned on the channel. Click "Close" button or minimize technique window.

7. If you want control ZIVE SP/MP electrochemical workstation using "Single channel control/monitor", you can see following windows.

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To start experiment, click Start button

If you want control ZIVE SP/MP multichannel, you can see the following window

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on the channel. If you want to start experiment for multiple channel,

click on Group column which you want to start and click Start button at bottom side.

8. After click start button, you can see the following box.

To start experiment for specific channel, click Start button



The data file name is automatically determined as default value but if you want to change data file folder then click





You can make new folder which you want to save.

- If you want use your file name, input your file name on "File name"
- If you did not make file name manually, new data folder which named as sequence file name or technique file name under data folder and it's subfolder will be created with name as date. The data file will be saved in this folder. The data file name will be determined automatically as string with sequence file name or technique file name and hour and minute and second, finally channel number will be added.

example) If you used sequence file for test and its file name is testabc.zsc and channel number is 1 and test date is 2000 year January 1<sup>st</sup> time is 15:30: 1 then the data file will be saved as

C:\Zive data\sm\data\testabc.zsc\2000010115\3001\_01.sdo (If data file have DC data only)

Or

C:\Zive data\sm\data\**testabc.zsc\2000010115\3001\_01**.sde (If data file have DC data and EIS data) Or

C:\Zive data\sm\data\testabc.zsc\2000010115\3001\_01.seo (If data file have EIS data only)

e.g.) schedule file name with extension\yearmonthdayhour\minutesecond\_channel number If you input file name as "tommy" then

C:\Zive data\sm\data\testabc.zsc\2000010115\tommy3001\_01.sdo (sde,seo)

- 0  $\quad$  Start Step: If your sequence file has several steps, you can select starting step.
- ② Start file: If batch file is loaded, select file number
- ③ Current polarity: If you want reverse polarity for measured current, check on this box.

- ④ Active area: For calculation of current density
- (5) Capacity: For C-rate value calculation
- (6) Weight: For specific capacity calculation
- ⑦ Tester: Operator name
- ⑧ Memo: Text information for test (For line change, use CTRL+ENTER)
- No Save file: If you click this check box, the data will NOT be saved.

If you are ready to test, click OK button then experiment will be started.

You can see real time plot as the following. When experiment done, the display will be changed as following.



Constant voltage step display

 $\geq$ 



EIS measurement step display

Real time EIS graph shows sub window at right upper side. This window display sine wave or E vs. I graph to check data quality. Default display is sine wave.



This shows two sine wave graphs. One is for current (green line) and the other is for voltage (Yellow line). If you switch this graph to I vs E, double click on the graph then graph format will be changed.



Each graph will be overlayed by step voltage change.





 You can display graphic by clicking button so tool bar Click "Open data file" icon and select data file which you run the experiment.

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SM can split each EIS graph by selecting "step" in "division".

You can split data by file, cycle or step. Splitted data set's color was changed and graph line will be broken

ZMAN will split each EIS data automatically if you insert "cycle" mark.

For user defined graphic, you can select X axis and Max 4 Y axis parameter as you wanted.



#### **Data Analysis**

3.

- 1. Open "ZMAN" by clicking ZMAN icon. ZMAN program is located c:\program files\zivelab\zman2.2 folder.
- You can see the following window. 2.





Select Change Base folder and select the folder which contain SM data.

#### Select data file by double clicking 4.



If you inserted Cycle market as we used the above sequence file, above box will appear. Click "Yes" then multiple EIS data will be split automatically.



If you do not want to use all EIS data set, Click "No" then you can see select box.



You can select one EIS data for analysis.

5. Parameter for voltage name can be changed from X1 to Voltage for example by clicking "edit item" button

(1) 2952_01_1 (2) 2952_01_2 (3) 2952_01_3 (4) 2952_01_4 (5) 2952_01_5 (6) 2952_01_5 (7) 2952_01_7 (8) 2952_01_8 Rename	1st Control Parameter Voltage 2nd Control Parameter X2 3rd Control Parameter X3
(1) 2952_01_1	

6. You can input each voltage value for each EIS data using X1 colum.



7. You can see Impedance, Admittance, Modulus, Dielectric Constant by selecting as the following a) Select data set



b) Seelect Impedance,Z in polar,Admittance,Y in Polar, Modulus, M in polar, Dielectric Constant, E in polar. (in polar = in Polar Coordinate)



#### 8. Click 2D nyquist plot



You can see "Matching scale" by clicking right button of mouse and select "Matching scale"



Matching scale is X axis scale and Y axis scale on graph is same. You can check each data value by selecting "cursor" with clicking right button of mouse .

Show	🕨 🗸 Legend
Remove Bad data	Palette
Rescale to Show All Matching Scales	✓ Impedance, Z Admittance, Y
Bring Cursor to Center Go to Cursor	Modulus, M Dielectric Constant, E
Items	✓ Only Raw Data
Contents	Raw + KK
Axis	Raw + Fitted
Export Image to Clipboard	



You can move the point by dragging the mouse or by keyboard arrow key .

9. Click 2D Bode



### **EIS Anaysis**

- Select Modeling on Analysis
- 1. Select data

Choose one data set having more complicated shape



2. Search Model by clicking "search" button

Target Categories		
CONDUCTING POLYMER (20) CORROSION COATING (31)		
ECHEW (7)	Sector Sector	Ū.
FUEL CELL (32) METAL ELECTRODE (23)		
	R Remove Duplicat	15
Farameters		
Free but must stay positive		
Initial Guessing		
Method	Simple Circular	
Nonlinear Fitting		
Data Sequence	Z. Real & Imag	
Weighting Facto	Modulus to Data	
Stepare	El off	
Do you want to sta	rt searching process?	

- 3. Select Battery & Supercapacitor in Target category
- 4. Select Parameters range as Free but must stay positive
- 5. Select initial guessing method as simple circular
- 6. Select data sequence as Zreal & Zimg
- 7. Select weight factor as modulus
- 8. Click Yes button then automatic model searching will be done



Result was rearranged by chi square value order.

- 9. Check on Bisquare for more accurate searching.
- 10. Click Start button and press stop button about 10<sup>th</sup> model searching finished or until end of similar fitting overlay plot on Nyquist & Bode..



searching result will be rearranged by chi square value and it may be different from previous result.

- 11. If the error level per parameters are too high, you can search again with other weighting factor.
- 12. By clicking you can change searching parameters. Select weighting factor as unit

Target Categories	pories	×		
raiger caregoires				
CONDUCTING PO	DLYMER (20)			
CORROSION COM	ATING (51)			
ECHEM (7)		8		
FUEL CELL (32) METAL ELECTROD				
METAL ELECTRON		-		
	Remove Dup	olicates		
Parameters				
Free but must r	stay positive			
Initial Guessing				
Method	Simple Circular			
Nonlinear Fitting				
Data Sequence	Z: Real & Imag			
Weighting Facto	ar Linity			
Bisquare	C Off			
	nt l'us l'us reas	187		
Net O Her				Res 2 Ant fo
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Start O the Intel Gaussing C by Simple any Model Ministry An-Aming Ministry An-Aming	Chule C by Leven And Chule A 7 6840-0 5 (45-44) = 10551-0 11 (45) = 10551-0 1	18- 191 - 12-		Ann D Sea fr
Bark O The India Gaussing City Single my Model Mith & Gaussing City City Mith & Gauss City Mith	Chule () by leven And () by ap +7 () by ap +7 () by ap +1 () by ap	18- 18- 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- <u>5</u> 10- 10- 10- 10- 10- 10- 10- 10-		Ren D Ben fi
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Bat         Train	Ortubar         by Gamma         Sec           (3)-app         #         (3)-app         #           (4)-app         #         (3)-app         #           (4)-app         #         (3)-app         #           (4)-app         #         (4)-app         #           (4)-ap		sis 15 15	4 28 28 38 2 204
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	Cincler 1 by Gaussi Anno Cincler 2 by Gaussi Anno (Cincler 2 b) (Cincler 2 b)		a a a	4 2 10 2 10 10
	Cincler 1 by Gaussi Anno Cincler 2 by Gaussi Anno (Cincler 2 b) (Cincler 2 b)			4 2 10 2 10 10

- 13. Repeat previous 9-10 steps.
- 14. If top listed model have parameters which needs lock, then lock parameter and fit again.



Suggested 1<sup>st</sup> model by automatic model searching is Rs-C1|(C2-Q1)|(C3-Q2)|(R1-W)



- 15. Check on "ALL" and click fitting button for series fitting
- 16. If there are data sets having singular matrix after series fitting, click initial guessing(simple circular fitting) and click fitting button until showing normal result (no singular matrix) for each data set. If singular matrix remains, try to use genetic analysis as initial guessing and click fitting

To compare each element(parameter) for series measurement data sets, do not lock parameter anymore.

17. If all data set fitting result are normal, Click select button to take this model



- 18. You can get this result or do fitting again by clicking fitting button.
- 19. If you want to accept this result, click OK button
- 20. If you want to choose other model, click right mouse on searching button



21. Click Show previous result pop up menu



22. Select Minimum parameters on the top list. It is Rs-(R-W)|Q



23. Check on Bisquare option and click fitting button



- 24. Do step 15-18 again.
- 25. If you accept this result, click OK button
- 26. If you want overlay raw data and fitted data, Plot detail in Plot menu



• You can see Nyquist or Bode data with fitted data



• 3D graph





Surface plot (Nyquist)

Surface plot( Bode, Phase)



Surface plot(Bode, impedance)

Surface plot(Admittance)

• To view parameter plot, Select Plot-2D parameter



Select parameter(s) and click apply button





Qy vs time

Qy vs. time





R1 vs. voltage

R2 vs. voltage



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