ModuLab®

electrochemistry

Application Guide

DC cell analysis techniques
Pulse Voltammetry

John Harper and Brian Sayers
Solartron Analytical, Farnborough, UK.

Application Guide: AGML11

Introduction

Polarographic techniques were among the first to be developed by electrochemists as a set of standardized methods with analytical solutions of diffusion equations. They are commonly employed in trace analysis applications such as trace metal analysis in water quality and analysis of blood. Historically, mercury electrodes were the substrate of choice. However, due to the toxicity of mercury, many studies now use inert materials such as boron-doped diamond electrodes. This demonstration experiment shows how the ModuLab potentiostat applies and measures the voltage and current for three common polarographic techniques; normal, differential and square wave pulse voltammetry.

Key system capabilities used in this demonstration

- In addition to conventional forward and reverse current measurements per pulse, ModuLab allows the user to sample current and voltage continuously while using pulse techniques. This allows the user to check the pulse shapes and decide where the “per pulse” type measurements should be made. This is a very useful feature since the timing varies due to the cell time constant and DC scan rates.
- This is also a useful feature to check that the correct waveform is being applied to the cell.

Equipment required for this demonstration

- ModuLab Potentiostat, ModuLab Test Cell

Connections

- Connect coloured cables to their corresponding connections on the test cell

Experiment setup

Select "AGML11 Pulse Voltammetry" in the "ModuLab Application Guide" project
Either a multiple step experiment as below, or individual experiments for each pulse technique

Step #	Purpose
Step 1	Normal Pulse Voltammetry step
Step 2	Differential Pulse Voltammetry
Step 3	Squarewave Voltammetry
Additional test possibilities: • The low current femto ammeter option may be used for better current resolution if needed.	

Notes on setup

Data is normally presented as the differential current vs. applied potential. To observe this simply select the graph file and change the axes to Diff. I vs. E (in the drop down menu). For each technique, two scans were measured. The first measured the current in ‘per pulse’ mode (in DC Measurement Setup window). The current is integrated over the last 10% of the pulse and this is the normal method that end user would select. The second step acquired data periodically so that one can see the applied voltage and measured waveforms. These results are discussed further in the text.

ModuLab has not been configured to use with dropping Mercury electrodes to date. Should this be of interest to your customer, please contact the Solartron representative for your region.

Data presentation and analysis

Figures 2 to 4 show the current and voltage waveforms for normal, differential and squarewave pulse voltammetry respectively. Precise applied waveforms and measurement of the response of the cell are critical for these techniques since one wishes to reduce the influence of charging currents. Failure to do so results in loss of sensitivity. It is clear from the results that accurate applied and measured waveforms are possible even at very short pulse periods as demonstrated in the squarewave voltammogram where the pulse duration is in the order of 10 ms.

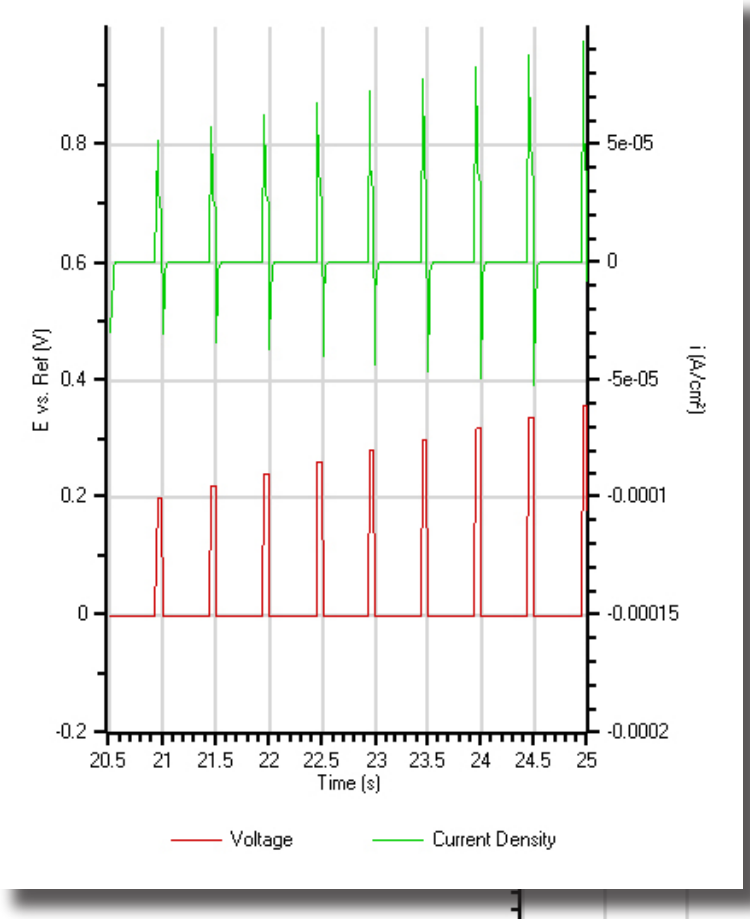

Figure 1

E and I vs. time, Normal Pulse Voltammetry

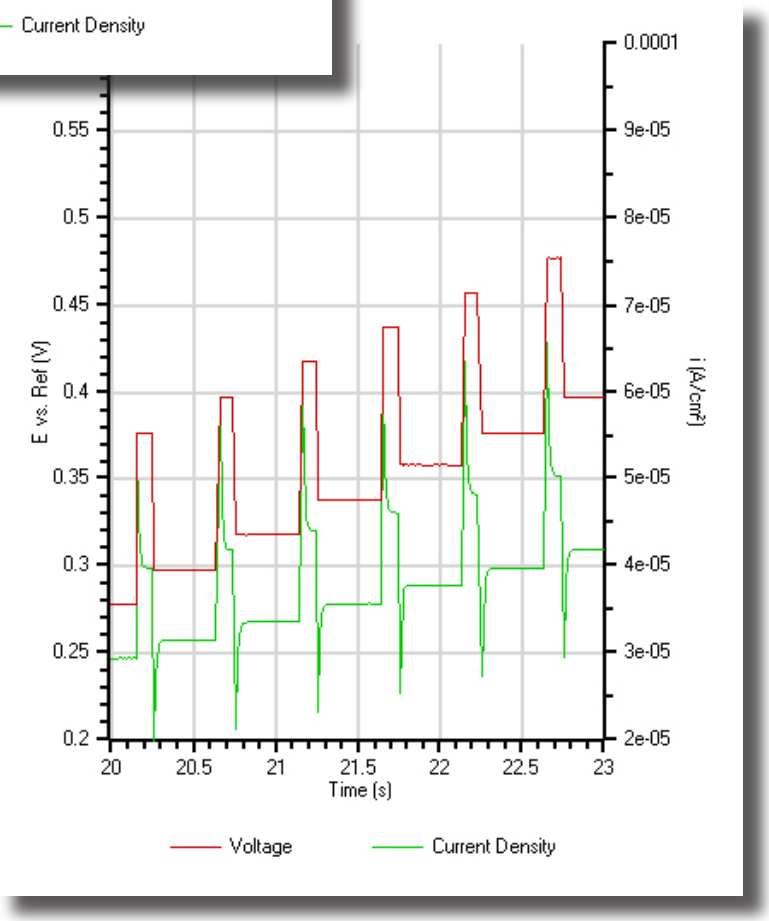

Figure 2

E and I vs. time Differential Pulse Voltammetry

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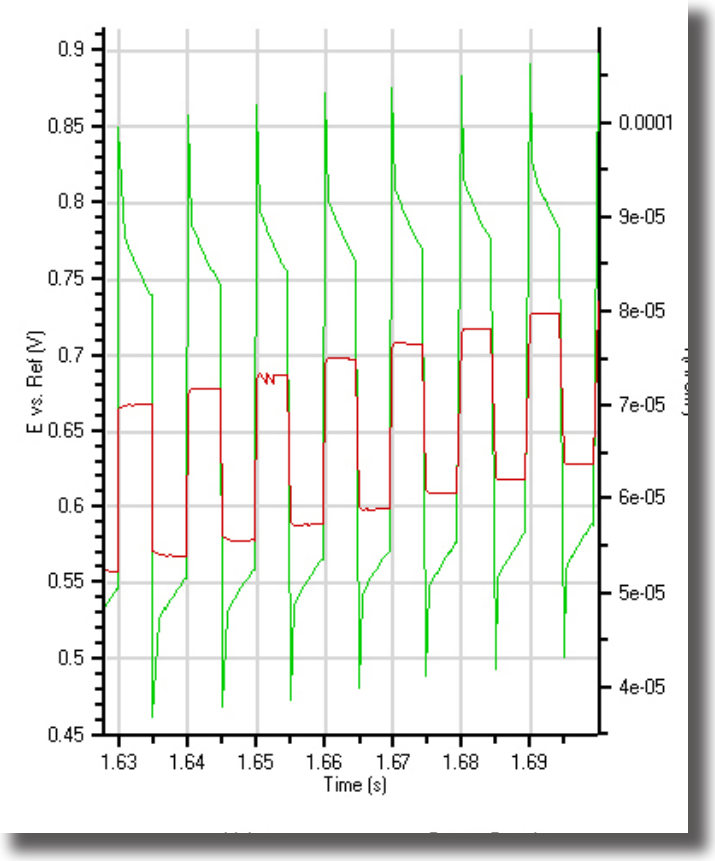

Figure 3

E and I vs time, Square Wave Voltammetry. Note that the pulse frequency = 100 Hz

Conclusions

The ability of the ModuLab software to measure applied waveforms continuously or at any point defined by the end user is a powerful feature. In pulse voltammetry and polarographic analysis this will be useful to check that the correct waveform is applied to the cell and that measurements are taken at the correct point on the current decay waveform.

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UNIT B1 ARMSTRONG MALL
SOUTHWOOD BUSINESS PARK
FARNBOROUGH, GU14 0NR
UNITED KINGDOM
Phone: +44 (0) 1252 556 800
Fax: +44 (0) 1252 556 899

801 SOUTH ILLINOIS AVENUE
OAK RIDGE
TN 37831-2011
USA
Phone: +1 865 425 1360
Fax: +1 865 425 1334

Visit our website for a complete list of our global offices and authorized agents

solartron.info@ametek.com www.solartronanalytical.com

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