

DC cell analysis techniques Fast scan voltammetry

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Introduction

Cyclic voltammetry is without doubt the most commonly used technique in the electrochemical sciences. Fast scan cyclic voltammetry uses very high scan rates to probe fast electrochemical processes and scans typically above 10 V.s⁻¹ to tens of kV.s⁻¹ are used. The fast data capture rate of the ModuLab potentiostat coupled with a fast slew rate (approximately 10 million volts per second), allows the user to measure ultra fast processes on microelectrodes. This will be of particular interest to those researching bio-electrochemical applications such as determination of the concentration of Dopamine in the brain and other neurological studies. This demonstration guide explains how to run fast scans up to 10 kV.s⁻¹ using the test cell. Scans up to 100 kV.s⁻¹ are included to demonstrate the measurement speed of the system.

Key system capabilities used in this demonstration

- Fast ADC's over-sample current and voltage up to 24 MS/s (24 million samples per second or every 42 ns!). ModuLab outputs data to the PC at up to 1 million samples per second. Therefore each point has been sampled 24 times for improved signal / noise and repeatability.
- Fast / smooth waveform generator (64 MS/s sample rate, capable of outputting waveform samples to the DAC every 16 ns). Produces ultra-smooth and precisely controlled waveform. All the benefits of analog smoothness with digital precision.
- Digital ramps offer benefits over analog including ability to apply very slow sweep below 10 mV.s⁻¹, improved applied voltage accuracy and no time drift, often observed with analog generators.

Equipment required for this demonstration

• ModuLab potentiostat, ModuLab test cell.

Connections

 Connect the ModuLab potentiostat to the ModuLab test cell using the connection diagram shown in the following experiment.

Experiment setup

Select "AGML10 Fast Scan Voltammetry" in the "ModuLab Application Guide" project Either multiple step experiment as below, or individual experiments at each scan rate.

Step #	Purpose
Step 1	Cyclic voltammetry at 10 V/s
Step 2	Cyclic voltammetry at 100 V/s
Step 3	Cyclic voltammetry at 1 kV/s
Step 4	Cyclic voltammetry at 10 V/s
Step 5	Cyclic voltammetry at 100 V/s
Additional test possibilities:	

- Try different integration times to see the effect of integration on noise rejection
- Try different sample rates for capturing data
- Try using "E change" mode to capture data when the voltage changes by a set amount
- Set up a multi-cycle CV capturing every 10th cycle instead of every cycle to reduce the data storage requirement



Data presentation and analysis

Cyclic voltammograms using scan rates from 10 V.s⁻¹ to 100 kV.s⁻¹ are presented in Figure 1.

Important features to note include the contribution of the capacitance of the cell to the total current. According to theory, the current flowing through the cell capacitance increases with scan rate. This is significant in microelectrode studies since the reactance of the capacitance may become small in comparison with the charge transfer impedance. Indeed, if the impedance of the capacitor is insignificant compared with the charge transfer resistance, current will flow through the capacitor and therefore kinetics cannot be measured. It is for this reason that many fast scan experiments utilize ultra microelectrodes (UME's) as their double layer capacitance is small.



Figure 1 . For details of measurements refer to hardware settings in the setup file associated with this demonstration guide.



Figure 2. Voltage vs. Time representation of a fast scan cyclic voltammogram (forward scan only). Example shown was measured using a scan rate of 100 kV.s⁻¹ with data acquired at 1 million samples per second.

Figure 2 demonstrates the speed of data capture available with ModuLab. Such rates are necessary to accurately represent current - voltage characteristics of the cell under test. This is achieved by using fast sampling ADC's, capable of over-sampling current and voltage up to 40 times giving up to 1 MS/s data acquisition rate. The samples are averaged over the integration period or sample rate (which ever is the shortest) to produce an output that is accurate and with a high signal to noise ratio.

Summary

Advanced facilities such as fast data capture, smooth digital waveform generation and high slew rate electronics allow the user to perform measurements in excess of 10 kV.s⁻¹. Through the use of state-of-the-art digital signal processing technology and the latest generation fast sampling ADCs and DACs, the ModuLab system can overcome the limitations of traditional analog potentiostats without sacrificing performance associated with older digital potentiostats. In conclusion, the digital potentiostat can now apply smooth waveforms that are indistinguishable from analog ramps whilst overcoming performance limitations of analog ramp generators.



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