Application Note

# Measurement Techniques for Electrochemical Water-Splitting

## Application Note: 2023/01

Here we provide a brief overview of the standard measurement techniques that could be used to evaluate the properties of catalysts designed for electrochemical water splitting using the *Princeton Applied Research (PAR)* instruments.

**Introduction**

Electrochemical water splitting is the process of separating water into its two constituent elements, hydrogen, and oxygen, using electricity. This process involves the use of an electrolyzer, which is an electrochemical device that uses an external electrical current to drive a non-spontaneous chemical reaction.

The electrolyzer typically consists of two electrodes, an anode, and a cathode, separated by an electrolyte solution. When an electric current is passed through the electrolyte, the water molecules at the anode are oxidized to produce oxygen gas, while the water molecules at the cathode are reduced to produce hydrogen gas.

Oxidation at anode: $2H_2O(l) \rightarrow O_2(g) + 4H^+(aq) + 4e^-$

Reduction at cathode: $4H^+(aq) + 4e^- \rightarrow 2H_2(g)$

The overall reaction can be written as: $2H_2O(l) \rightarrow 2H_2(g) + O_2(g)$

This process is important to produce hydrogen as a clean energy source, as hydrogen can be produced through the electrolysis of water using renewable energy sources like solar or wind power. Electrochemical water splitting using proton exchange membrane (PEM) electrolyzers is an attractive approach for producing hydrogen as a clean energy carrier. Oxygen evolution reaction (OER) is an essential process in PEM electrolyzers that involves the transfer of four electrons, producing oxygen gas from water.

To develop efficient electrocatalysts for the OER, several performance metrics are typically used for evaluation. In this technical note, we will discuss the most common techniques used for evaluating electrocatalysts for OER. Here in these measurements, we used Nickel foam as a standard catalyst with 1M NaCl aqueous solution as standard electrolyte. All the results are without iR correction.

***1. Electrochemical measurements***

The most common technique for evaluating electrocatalysts for OER is electrochemical measurements. Electrochemical methods such as cyclic voltammetry (CV), and linear scan voltammetry (LSV). CV is used to determine the redox behavior of the catalysts, while LSV is used to obtain the electrochemical activity and the onset potential for OER.

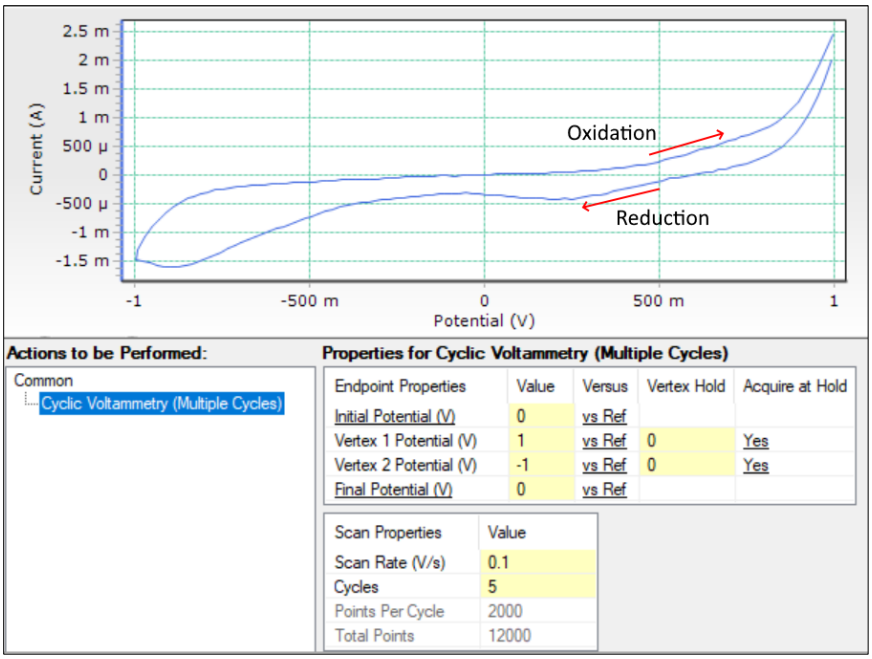


*Figure 1. Cyclic voltammetry in the potential range of -1 to 1 V (scan rate: 0.05 V $s^{-1}$).*

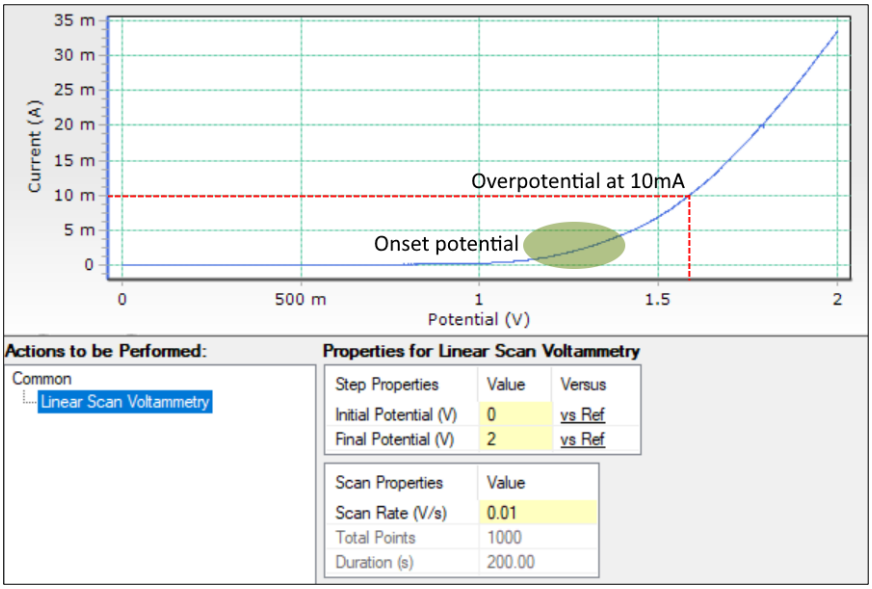


*Figure 2. Linear scan voltammetry in the potential range of 0 to 2 V (scan rate: 0.01 V s-1).*

## *2. Tafel analysis*

Tafel analysis is a method used to extract kinetic information about the electrocatalyst. The slope of the Tafel plot (logarithmic plot of the OER current density versus the overpotential) is related to the OER reaction mechanism and provides information on the rate-limiting step. A lower slope indicates faster kinetics.

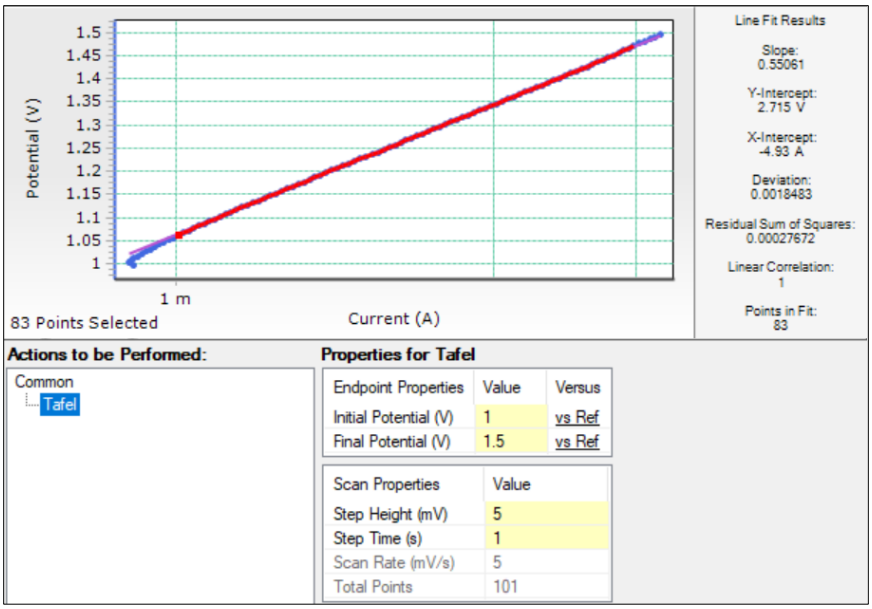


*Figure 3. Tafel analysis in the current range of 1-10 mA (current in logarithmic axis)*

### *3. Electrochemical impedance spectroscopy*

Electrochemical impedance spectroscopy (EIS) is a technique that measures the impedance of an electrochemical system as a function of frequency. EIS can provide information about the charge transfer resistance of the electrocatalyst and the double-layer capacitance. A lower charge transfer resistance indicates faster electron transfer kinetics.

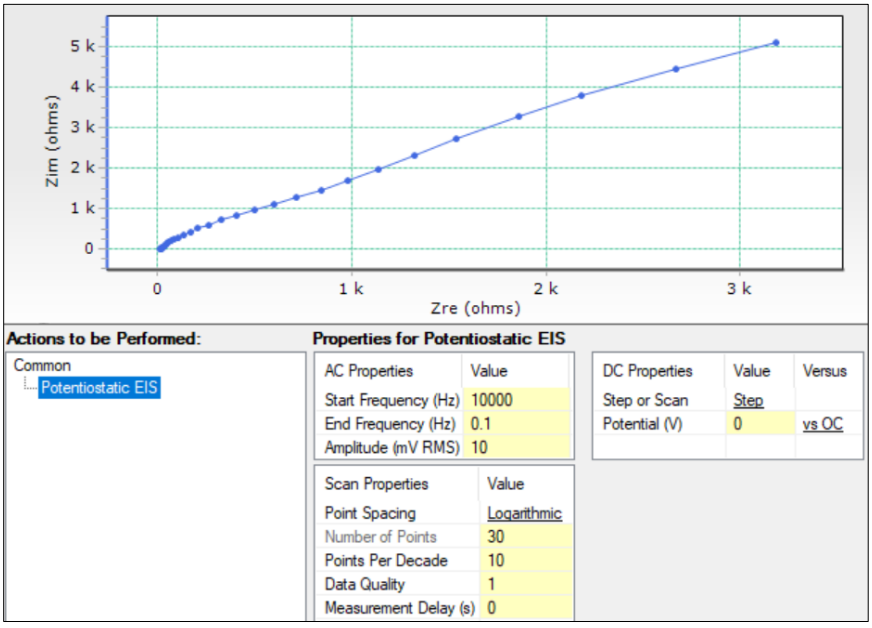


*Figure 4. Potentiostatic impedance measurements at OCV in the frequency range 10,000 to 0.1 Hz*

### *4. Durability testing*

Durability testing is crucial for evaluating the long-term stability of the electrocatalyst. Accelerated aging tests, such as continuous potential cycling and long-term stability tests, can be used to simulate the operating conditions of the electrocatalyst. For this purpose, Chronoamperometry (CA) can be used to measure the current density as a function of time.

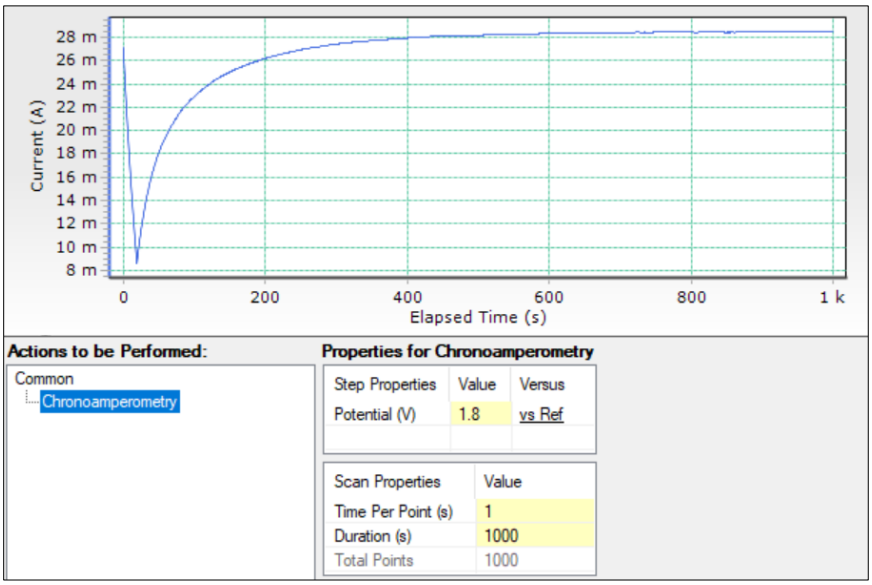


*Figure 5. Chronoamperometry (CA) measurements at a potential of 1.8 V.*

**Summary** – In conclusion, evaluating the electrocatalyst for electrochemical water-splitting involves a combination of measurement techniques, such as cyclic voltammetry, linear scan voltammetry, Tafel analysis, electrochemical impedance spectroscopy, and durability testing. These techniques provide valuable information on the electrochemical performance, kinetics, and long-term stability of the electrocatalyst. The instruments that support these techniques include AMETEK Princeton Applied Research (PAR) potentiostats and for upscaled water splitting we recommend high-power AMETEK Solartron Analytical instruments. Furthermore, for performing in-depth analysis of localized surface electrochemical behavior for catalyst films, techniques such as scanning electrochemical microscopy (SECM) can be used. SECM could be performed using AMETEK PAR VersaSCAN that allows non-destructive, high-resolution characterization of electrochemical systems, providing quantitative information and imaging capabilities to visualize electrochemical response.

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