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Electrochemical Impedance Spectroscopy for Characterizing Industrial Electrochemical Systems

The *Pulsenics Probe* is a first-of-its-kind solution that successfully applies the principles of *Electrochemical Impedance Spectroscopy* (EIS) to electrolysis-based processes operating in real-world conditions. The Pulsenics technology allows users to monitor the changing conditions of individual cells within a stack, including electrodes, membranes, and electrolytes: data that was previously unattainable at the industrial level. With this innovation, users gain an understanding of the electrochemical performance of their systems, leading to faster development, more confident quality assurance, and improved uptime in operations.

Solving the limitations of traditional EIS measurement instruments by introducing real-time diagnostics of electrochemical stacks in operation

Electrochemical Impedance Spectroscopy (EIS) is a powerful technique for characterizing electrochemical processes. By measuring the impedance of an electrochemical system across a range of frequencies, electrochemists gain valuable insights into their system's performance. These insights include information on the resistance and capacitance of the system's components, collected in a non-destructive and non-invasive manner. EIS is especially effective in isolating sources of performance loss, making it a valuable tool for the electrochemical industry. With its ability to isolate inefficiencies in the electrochemical process, EIS helps researchers and developers characterize their systems for maximum efficiency and performance.

Electrolyser technologies continue to scale to reach higher power densities and benefit from economies of scale. At these higher power levels, the electrical characteristics of the electrochemical stacks are affected in two main ways:

1. The stack experiences a much lower cell impedance compared to what is typically encountered at the lab scale; and
2. The stack is subjected to a wide range of conducted, emitted, and radiated noise in the industrial environment.

These effects can significantly compromise the quality of the EIS data and therefore must be carefully accounted for when performing EIS measurements at the industrial scale.

In addition, traditional EIS-capable instruments, such as potentiostats, were not designed to operate in demanding industrial environments. They were primarily designed to operate on lab-scale benchtop single cells running at low DC voltages and currents. Therefore, they have

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an inherent limitation that prevents their use as a reliable EIS measurement device for large industrial electrochemical stacks.

These limitations restrict the user's ability to develop models or analyze the data to gain insights into the system's performance once it is out of the lab. Therefore, performing EIS on electrochemical stacks operating at industrial power levels requires careful measurement considerations and dedicated hardware that is capable of handling the higher power levels while still providing accurate and consistent measurement over the full frequency range.

A high-quality dataset is a precursor to reliable data-driven decision making: A comparison between the Pulsenics Probe and a state-of-the-art potentiostat.

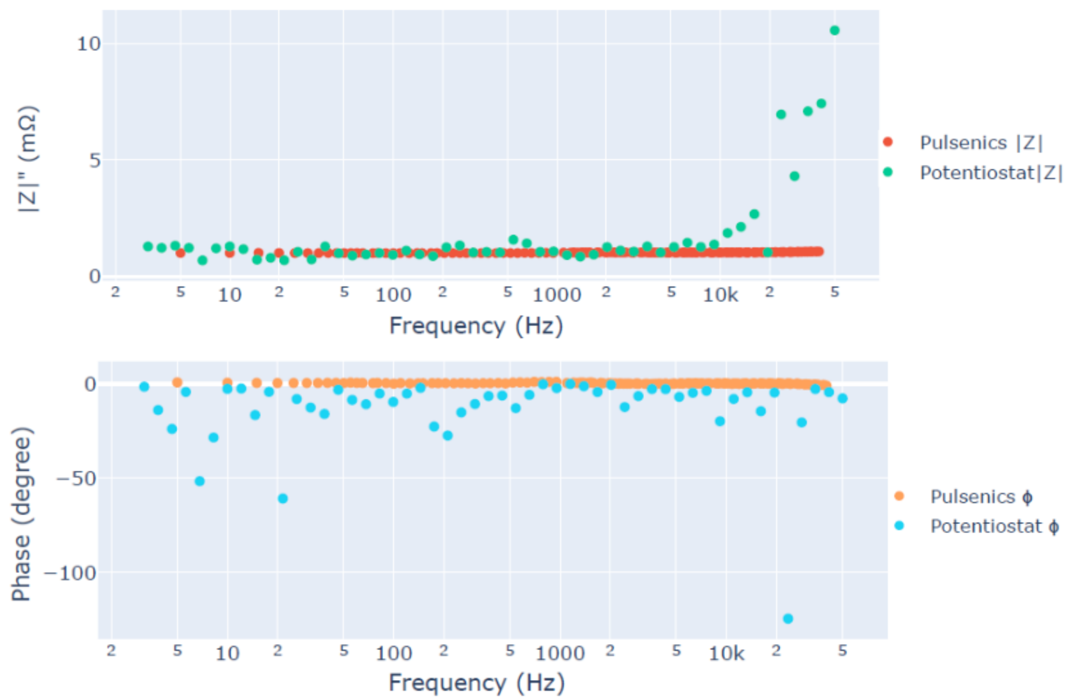

Figure 1: Comparison of Pulsenics Probe EIS with a leading potentiostat, on a 1 mΩ shunt calibration resistor.

At Pulsenics, we are obsessed with giving our customers the highest quality data, because we strongly believe this is a crucial first step towards any physics or model-based data analysis. **Figure 1** shows a comparison of the frequency domain response between a state-of-the-art high-voltage potentiostat and the Pulsenics Probe. For this setup, the customer used a 1 mΩ coaxial shunt resistor with a 1% tolerance up to the several MHz range. The 1 mΩ coaxial shunt resistor should have an impedance magnitude of 1 mΩ and a phase angle of 0° within the range of frequencies tested.

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As shown in **Figure 1,** both the Probe and the potentiostat produce good results for the impedance magnitude up to 1 kHz. However, the data collected by the potentiostat deviates significantly above 1 kHz frequency, while the Pulsenics Probe's data remains accurate with a less than 1% deviation from the expected value. The Probe also gave accurate phase angle values across the measured frequency bandwidth, while the potentiostat failed to produce consistent measurements throughout the entire frequency range.

Inaccurate phase measurements lead to unreliable Bode and Nyquist plots. In this case, for the phase angle data collected by the potentiostat to be usable, an unrealistic 35% error allowance would need to be accepted: an error that most operators and developers of electrochemical stacks deem impractical to work with. At Pulsenics, we are meticulous about the type and accuracy of the data our customers rely on.

The results of this demonstration were expected as potentiostats are not designed to measure very low impedance systems at high voltages. As a rule of thumb, most potentiostats operate accurately up to a frequency of $f_{trust}=R*10^3$ kHz, where R is the resistance in ohms. Hence, for the 1 mΩ resistor employed in this demonstration, the impedance magnitude data obtained from the potentiostat could only be trusted up to 1 kHz. However, as depicted in **Figure 1**, the phase angle data is only usable when a significant percentage of error allowance is acceptable.

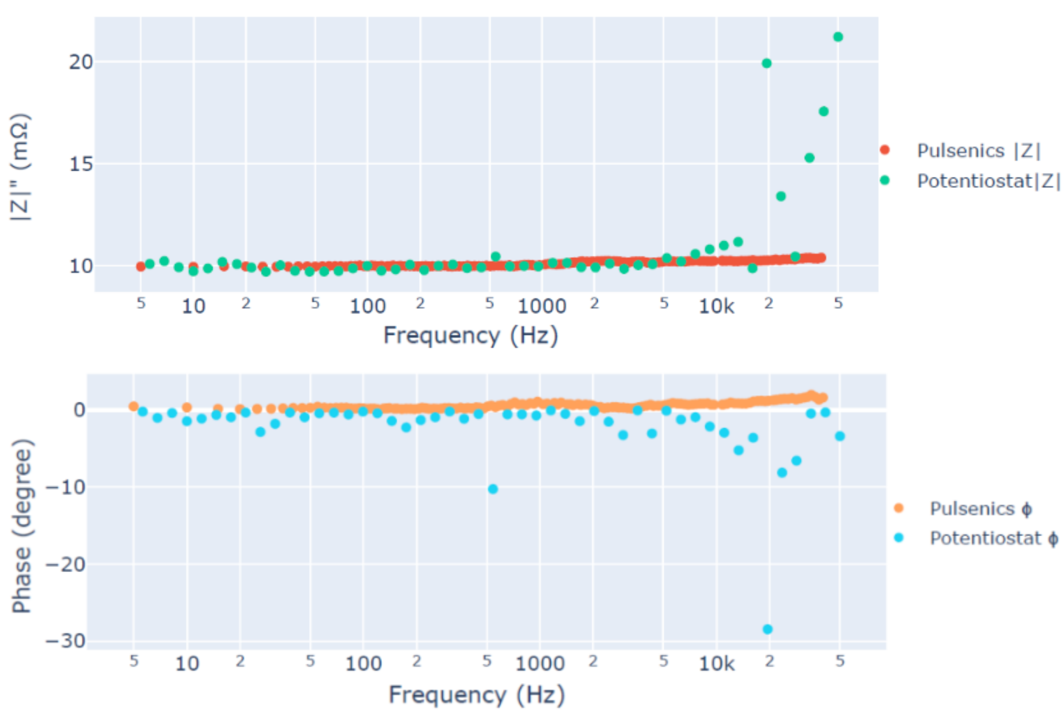

Figure 2: Comparison of Pulsenics Probe EIS with a leading potentiostat, on a 10 mΩ shunt calibration resistor.

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Figure 2 presents another comparison of the frequency domain response between the state-of-the-art high-voltage potentiostat and the Pulsenics Probe. However, for this comparison, the customer used a 10 mΩ coaxial shunt resistor with a 1% tolerance up to the several MHz range. The 10 mΩ coaxial shunt resistor is expected to have an impedance magnitude of 10 mΩ and a phase angle of 0° within the range of frequencies tested.

As depicted in **Figure 2**, both the Probe and the potentiostat produce good results (within 1% error) for the impedance magnitude up to 5 kHz. However, as with the 1 mΩ shunt test, the potentiostat impedance magnitude data deviates significantly beyond the 5 kHz range, while the Pulsenics Probe remains accurate with a less than 1% deviation from the expected value across the entire frequency range tested. For the phase angle values, the potentiostat data began to deviate significantly above 6 kHz and included an outlier (500 Hz), while the Pulsenics Probe gave accurate (within 1%) results across the entire tested frequency range.

The results of this demonstration show that at slightly higher impedance values, the potentiostat's performance improves, yet it is only able to accurately measure impedance within a narrow frequency range. Despite the f_{trust} equation indicating that the potentiostat should have provided reliable data up to 10 kHz for a 10 mΩ resistor, the potentiostat only provided acceptable values up to 6 kHz. On the other hand, the Pulsenics Probe delivers accurate impedance measurements across the entire frequency bandwidth tested, up to 40 kHz and beyond.

Prior to analyzing the data on our software platform, Pulsenics supports customers in validating their datasets by running strict verification routines like the demonstrations that produced results from **Figure 1** and **Figure 2**. This step is crucial to remove any noise, faulty measurements, or potential bias from the final usable dataset. To ensure the quality of the data before deploying the EIS solution, our team performs verification routines that ensure the accuracy and consistency of measurements across the entire frequency range for spectroscopy over a wide range of operating conditions. By taking these steps, users can be confident that they are obtaining accurate and reliable results.

A high-quality dataset is not a luxury, especially as electrolyser technologies continue to scale to reach higher power densities. As previously mentioned, the challenging operating conditions of the electrochemical stacks experiencing low cell impedance, high current densities, and in the presence of noise, warrants careful consideration to proper EIS measurements. A common pitfall we see is the lack of proper detection, isolation, and accounting for noise in the system, especially with lower cell impedance levels. This significantly affects the validity of the generated models and insights and is a reason why developing good practices to obtain and validate high-quality datasets is integral for effective stack characterization.

Equipped with the Pulsenics Probe, engineers and researchers can accurately measure the impedance values of electrochemical systems, even at industrial power levels. Moreover, the Pulsenics Probe can measure impedance values for cells with very low impedance, surpassing leading potentiostats in this regard. These capabilities make the Pulsenics Probe the go-to standard for characterizing high-power electrochemical systems.

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ABOUT PULSENICS

Pulsenics' mission is to provide the tools to drive the electrochemical industry towards a more sustainable and efficient reality. More information is available at pulsenics.com.