

Quick Check of EIS System Performance

Introduction

The maximum frequency is an important specification for an instrument used to perform Electrochemical Impedance Spectroscopy (EIS). The majority of EIS manufacturers specify a maximum frequency of 1 MHz. Some manufacturer's specifications, however, misrepresent true system performance. Many EIS instruments specified to measure impedance at 1 MHz can apply a 1 MHz signal and measure a response -- but have huge errors when they do so.

This document is a protocol to quickly assess the performance of any EIS instrument. You can use it as an easy and effective comparison between systems. You should test before you buy!

The test routine involves measuring five EIS spectra. Three spectra are recorded using resistors with ideal EIS response. Two other spectra probe the limits of an EIS system. One spectrum is recorded in potentiostatic mode on a cell that approximates infinite impedance. The second spectrum is recorded in galvanostatic mode on a cell with close to zero impedance.

Gamry Instruments will be happy to provide three $\pm 1\%$ accurate resistors and a piece of braided copper for the near-zero impedance test. Simply call or email us and ask for the "EIS Test Resistors".

All high quality EIS systems should allow 4-terminal connections to the cell. In this common scheme, four leads are connected to the sample being tested. Two leads apply current and two leads measure voltage. Impedance is calculated by dividing the measured voltage by the current. EIS spectra cannot be measured accurately without a 4-terminal connection.

What do you do if your system cannot make 4-terminal connections? Connect the Reference lead to the Counter electrode lead in any convenient manner. Don't expect high quality results.

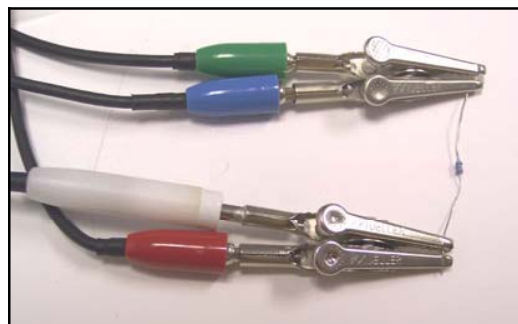
This document contains detailed cable positioning information. Improper connections to a resistor will add capacitive or inductive impedance to the ideal resistive impedance. If you follow the cable positioning shown here, the resistor spectra should have less than 2° of phase shift up to 1 MHz.

1 k Ω Resistor

The 1 k Ω resistor is marked with 3 brown and 2 black colored bands --

 (brown, black, black, brown, brown).

Hooking up to the 1 k Ω resistor is easy -- see the figure below. Connect the Working and Working Sense leads to one side of the resistor and the Reference and Counter leads to the other side. Keep the leads about 4 cm apart to minimize capacitive coupling between the leads.



Gamry's cell cable color codes are shown in the table below.

<u>Color</u>	<u>Lead</u>	<u>Function</u>
Green	Working electrode	current flow
Blue	Working Sense	voltage sense
White	Reference electrode	voltage sense
Red	Counter electrode	current flow

Measure the resistor's EIS spectrum using 20 mV_{rms} AC and zero DC voltage. Test frequency should extend from 0.2 Hz to the system's maximum frequency.

100 Ω Resistor

The 100 Ω resistor is marked with 3 black and 2 brown colored bands –

 (brown, black, black, black, brown).

Lead placement is critical for a non-inductive spectrum of the 100 Ω resistor. The Reference and Working Sense leads should be twisted together and attached very close to the resistor's body. The Counter and Working leads are also twisted together. They approach the resistor from the other side as shown below.



Measure the resistor's EIS spectrum using 20 mV_{rms} AC and zero DC voltage. Test frequency should extend from 0.2 Hz to the system's maximum specified frequency.

10 kΩ Resistor

The 10 kΩ resistor is marked with colored bands –

 (brown, black, black, red, brown).

This is the only resistor with a red band.

Connect the Working and Working Sense leads to one side of the resistor and the Reference and Counter leads to the other side. Keep the leads far apart to minimize capacitance across the resistor. We have measured 0.25 pF capacitance between alligator clips 10 cm apart -- enough to cause a 0.9° phase error at 1 MHz.

The figure below shows a very low capacitance connection.



Record the potentiostatic EIS spectrum of this resistor with zero DC and 20 mV_{rms} AC voltage. The frequency range should extend from 0.2 Hz to the system's maximum specified frequency.

Open Lead Test

The ideal cell for this test has infinite impedance. A large air-gap between the cell leads approximates this ideal.

Connect the Working and Working Sense leads together and place them inside a Faraday Cage. The Cage must be connected to the potentiostat's ground. Connect the Reference electrode and Counter leads together. With a Gamry EIS system, these leads can be left outside of the Faraday Cage.

With other systems, the Counter and Reference electrode leads outside the Faraday Cage may cause excessive noise. If this is true, move these leads inside the Cage. The separation between the pairs of leads should be at least 20 cm.

Record the potentiostatic EIS spectrum of this open lead configuration. We recommend testing with 50 mV_{rms} AC amplitude and zero DC voltage. The frequency range should extend from about 25 mHz to the system's maximum frequency.

Shorted Lead Test

The ideal cell for this test is zero impedance. The cell leads shorted together approach this ideal. In shorted lead tests the placement of the cell leads and the way the leads are connected is critical.

Clipping the cell leads to a piece of braided copper gives you a good low impedance connection. Clipping the alligator clips to each other always gave us more than 500 μΩ of contact resistance.

Twist the Reference and Working Sense leads together and clip them to one side of the braided copper. Twist the Counter and Working leads together and clip them to the opposite side of braid. See the figure below for the lead placement.



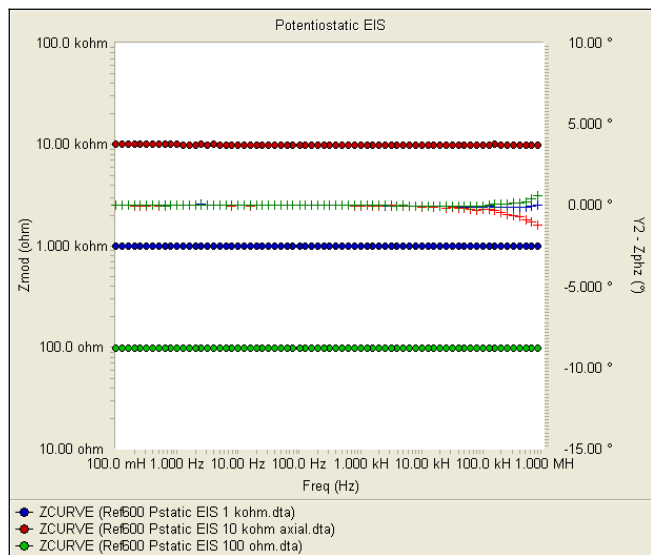
The Working Sense and Reference leads are the inside pair of connections. Don't allow the clip leads to touch except at the braid connections.

Note: Shorted lead measurements must be made using galvanostatic cell control. Potentiostatic mode does not work. If your EIS system does not offer galvanostatic mode – skip this test!

Record the galvanostatic EIS spectrum of the shorted lead configuration using an AC current that is about 1/2 of the instrument's rated current. Use zero DC current. The frequency range should extend from 0.1 Hz to about 20 kHz.

Resistor Results

The Bode Plot below shows EIS spectra of the three resistors measured with a Gamry Reference 600 Potentiostat. The circles are magnitude and the crosses are phase.

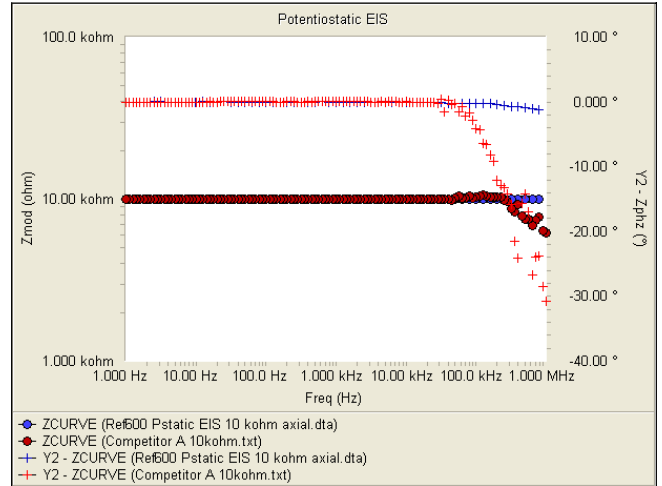


The magnitude of an ideal resistor's impedance is independent of frequency and the phase is zero.

In the spectra above, the magnitude variation is less than ± 1% and the phase angle is less than ± 2° at all frequencies.

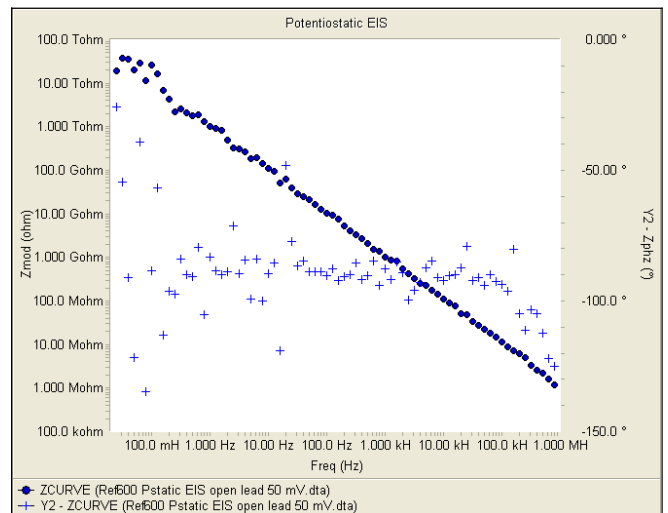
The following figure shows spectra recorded on EIS systems from different vendors. The cell was 10 kΩ in both tests. Again, the circles are magnitude and the crosses are phase.

The spectrum in blue was measured using a Gamry Instruments Reference 600. The red spectrum was recorded with a poor EIS system. This EIS system claims operation at 1 MHz. At 1 MHz, the errors are greater than 40% and 30°. This is unacceptable.



Open Lead Results

The open lead impedance represents the highest impedance that an EIS system will measure. All spectra measured on real-world samples consist of the sample's impedance in parallel with the open lead impedance. When you compare open lead spectra recorded on different EIS systems, higher impedance is better.



The Bode Plot above is an open lead spectrum measured using a Gamry Reference 600 Potentiostat. Over a wide frequency range, this looks like the spectrum of a small capacitor.

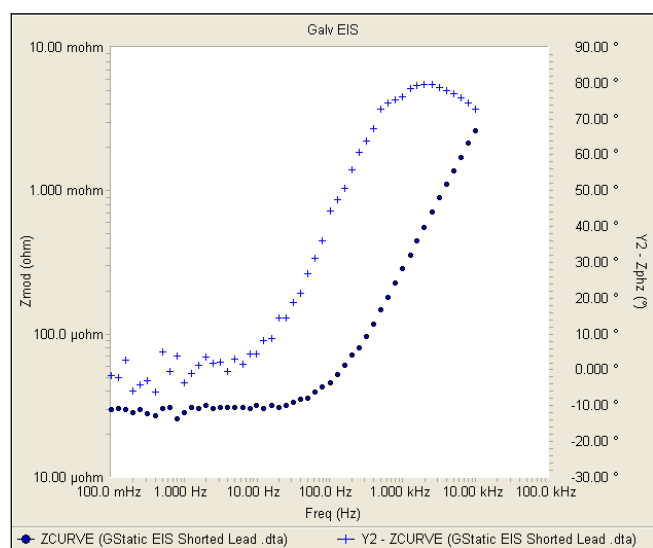
A capacitor model was fit to the region between 0.1 Hz and 100 kHz. The calculated capacitance was $0.16 \text{ pF} \pm 0.0004 \text{ pF}$.

This system's impedance at low frequency exceeds $10 \text{ T}\Omega$ ($1 \times 10^{13} \Omega$). Many EIS systems have low frequency open lead impedance below $100 \text{ G}\Omega$. This will severely limit their ability to measure high impedances.

Shorted Lead Results

Shorted lead impedance is the lowest impedance that an EIS system can measure. All spectra measured on real-world samples consist of the sample's impedance in series with ideal shorted lead impedance. When you compare shorted lead spectra recorded on different systems, lower impedance is better.

The Bode Plot below is a shorted lead spectrum measured using a Gamry Instruments EIS system with a Reference 600 Potentiostat. The cell leads were clipped to a piece of copper braid. The AC current was $300 \text{ mA}_{\text{rms}}$.



This spectrum fits well to a series RL model with a resistance of $30 \mu\Omega$ and inductance of 42 nH . The resistance value is interesting. The copper braid used to make the shorted connection has a measured

resistance of $8.4 \mu\Omega$ per mm. $30 \mu\Omega$ corresponds to a path of 3.6 mm through the braid.

The 3.6 mm resistive path seems reasonable.

This shorted lead spectrum was measured with the standard 60 cm cell cable provided with every Reference 600 Potentiostat. Significantly lower inductance will be measured using Gamry's special Low Z cable for the Reference 600.

The shorted lead test illustrates some of the experimental difficulty in measuring very small impedance. You must carefully separate the current paths and sense paths in your 4-terminal connection. You cannot ignore the resistance of metal connections.

Shorted lead curves measured on other EIS systems will probably look similar. The resistance and inductance values are likely to be higher than those from the above spectrum.

If you use shorted lead spectra to compare two EIS systems, make sure that you are comparing the two systems and not the quality of their shorted lead connections.

Conclusions

This test protocol can quickly check the function of an EIS system. It will quickly reveal marginal EIS systems, which typically exhibit very poor frequency response and unsatisfactory open lead and shorted lead spectra.

These tests also demonstrate the ability of an EIS system to measure very small or very large impedances. Knowledge of these limitations is very important. It can help you distinguish instrument response from chemistry.

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