

EIS for Interface-Zone Stability: Setup, Execution, QA, and Interpretation

(Stack-Agnostic Checklist)

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Table of Contents

- 1. Summary of Best Practices
- 2. Fast Checklist
- 3. Setup
 - 3.1. Test Fixture & Connections
 - 3.2. Calibration: Open/Short/Load Compensation
 - 3.3. Instrument Settings (Amplitude & Frequency)
 - 3.4. Environmental Controls
- 4. Execution
 - 4.1. OCV Stabilization & Pre-test Conditioning
 - 4.2. Pressure and Temperature Control
 - 4.3. Measurement Procedure & Monitoring
- 5. QA & Consistency Checks
 - 5.1. Kramers-Kronig Validation
 - 5.2. Repeatability and Drift Checks
 - 5.3. <u>Data Acceptance Criteria</u>
- 6. Data Interpretation
 - 6.1. Equivalent Circuit Modeling
 - 6.2. Avoiding Overfitting & Comparative Analysis
- 7. Traceability & Data Management
 - 7.1. Metadata and Logging
 - 7.2. Artifact Storage and Linking
- 8. Safety and Compliance Considerations
- 9. Limitations & Disclaimer
- 10. References
- 11. Appendices
 - A. Example EIS Parameter Table
 - B. QA Checklist Table
 - C. Glossary of Terms

Summary of Best Practices

Summary: Ensuring interface-zone stability via Electrochemical Impedance Spectroscopy (EIS) requires meticulous setup and consistent methodology. Use a **four-terminal guarded connection** to minimize parasitic inductance and capacitance, calibrate the fixture with open/short references, and apply a **small AC perturbation** (≈5–10 mV) to

maintain linearity 1 2. The cell stack should be at **open-circuit (steady-state)** and under controlled **pressure** and **temperature** conditions during measurement. Always perform **Kramers-Kronig (KK) consistency checks** on the impedance data to validate measurement integrity, and repeat tests to gauge reproducibility. Interpret results using simple **equivalent circuits** (R, CPE, Warburg elements) but avoid overfitting – additional circuit elements can improve fit mathematically while lacking physical meaning 3. Maintain thorough **traceability**: link each impedance dataset to its cell lot, test conditions, and environmental logs (temperature, humidity), and archive raw data (CSV) plus plots. Adhere to relevant **standards and safety guidelines** (e.g. use of IEC-prescribed 1 kHz AC impedance for quality control 4) and prioritize in-house safety protocols. The following checklist and detailed sections provide a step-by-step guide from setup through data interpretation.

Fast Checklist

- Fixture & Wiring: Use a 4-terminal (Kelvin) connection for the cell stack. Keep current-carrying leads twisted together or in coaxial configuration to cancel magnetic fields, and route sense leads separately to avoid inductive coupling 5 6. Secure all contacts (clean, tight clips) and place the cell in an earthed Faraday cage if measuring high impedances (to shield 50/60 Hz noise) 7
- **Guarding:** Connect the electrometer's guard terminal (if available) to a shielding plate or cable screens to shunt stray capacitive currents away from the measurement path ⁹ ¹⁰. Avoid ground loops (use the instrument's ground as reference).
- Open/Short Calibration: Perform open-circuit compensation with the cell fixture empty (terminals spaced as in a real test) to measure and null stray capacitance 11. Perform short-circuit compensation by connecting the fixture leads together (or using a calibration short) to null residual series resistance/inductance 12 13. Include a known load (resistor) calibration if the system supports full open/short/load correction.
- Instrument Settings: Start with a small AC voltage amplitude (≈5 mV rms, or 5–10 mV peak-to-peak) ¹⁴ at the cell's OCV. This ensures pseudo-linear response of the electrochemical system ¹. Caution: Too low amplitude may cause poor signal-to-noise (especially at low frequencies), while too high amplitude can drive the cell into non-linear behavior (manifested as distorted, harmonic responses) ¹⁵. Verify that no DC bias (other than OCV) is applied unless intended.
- Frequency Range: Choose a broad frequency sweep (e.g. 100 kHz or 1 MHz down to 0.1 Hz or 10 mHz) to capture all relevant processes. High frequencies probe inductive leads and ionic conduction; mid frequencies capture interfacial charge-transfer; low frequencies reveal diffusion.

 Note: For large high-capacity cells, frequencies around 100 Hz may yield more stable internal resistance readings than the traditional 1 kHz, due to reduced inductive influence 16. Very low frequencies (<0.1 Hz) greatly increase test time and risk of drift 17, so use them only if necessary.
- OCV & Stabilization: Allow the cell (or stacked sample) to relax at open-circuit until it reaches a steady state. Confirm that the OCV change is minimal (e.g. <△0.1 mV per minute) before starting EIS. Caution: An EIS measurement can take hours; any drift (e.g. continuing side reactions, gradual interface changes) during the scan will invalidate the linear time-invariant assumption and distort the spectrum 17.
- **Temperature Control:** Perform EIS in a temperature-controlled environment (e.g. 25 ± 1 °C or as required). Use a climate chamber or a thermal enclosure around the cell. Record the actual temperature of the cell/fixture. Temperature fluctuations or spatial gradients can lead to impedance changes during the test, compromising data quality.
- **Pressure Control:** If the interface of interest is pressure-dependent (as in solid-state stacks or pressed electrodes), apply a consistent stack pressure using a calibrated fixture (e.g. a spring-

loaded frame or hydraulic press). For example, tests on Li-ion pouch cells under controlled compression (0.27–2.7 MPa) used a stepwise pressure application with 20 min holds before EIS

18 19 . Ensure the pressure is maintained constant throughout the impedance measurement.

- Equilibration Time: After any change in state (assembly, pressure change, temperature change, or SOC adjustment), wait an appropriate period (e.g. 15–30 min for minor adjustments; several hours for major changes) before running EIS to let the system equilibrate. For instance, after clamping a cell in a pressure jig, a 20 minute stabilization was used before each EIS sweep in one study 18.
- Acquire Reference Spectra: Optionally, run a short test sweep on a dummy cell or known reference (like a resistor or RC network) to verify the instrument is functioning and the phase accuracy across frequencies. This also helps identify any systematic anomalies in the fixture setup before testing actual samples.
- Run EIS Measurement: Execute the frequency sweep, generally from high frequency to low frequency (to minimize time at the end when drift is more likely). Use an appropriate number of points per decade (e.g. 5–10 points/decade commonly) and integration time or averaging that balances speed with data quality. Monitor the live Lissajous (voltage–current) curves at a few frequencies if the instrument software allows, to ensure the response remains sinusoidal (no distortion).
- Real-time Monitoring: During the run, observe the Nyquist plot accumulation if possible. Look for tell-tale signs of issues: a sudden jump or loop might indicate a temporary disturbance or a poor connection. If such issues appear, consider pausing or aborting the test to investigate, as it's better to resolve mid-test than to finish a flawed spectrum.
- Post-Test Consistency: After the sweep, note the cell's OCV again. It should be nearly the same as before the test (deviations might indicate self-discharge or drift during measurement). If a significant OCV shift occurred, treat the data with caution the system may not have been in true steady state.
- Kramers–Kronig Check: Subject the impedance data to a Kramers–Kronig consistency validation (many EIS analysis programs offer this). This mathematical test checks if the data's real and imaginary components are mutually consistent for a causal, linear, stable system. Minor deviations are normal, but large inconsistencies mean the data is unreliable 20 21. Note: If the frequency range was truncated (not covering down to ~0 Hz or up to very high frequencies), the KK test may flag errors even for a stable system 22 23. In such cases, interpret KK results carefully or use a validated extrapolation model to fill in endpoints for the test.
- **Repeatability:** If time permits, repeat the EIS measurement under the same conditions to check repeatability. Two consecutive spectra on a stable cell should overlay closely (e.g. key resistance values within a few percent, no major shape changes). Consistent repetition builds confidence that the data reflect the cell properties and not random fluctuations. If repeats diverge, investigate possible causes (temperature drift, drying of electrolyte, instrumentation range issues, etc.).
- **Data Recording:** Save all raw data in open formats (CSV/TXT) with clear file names (include date, sample ID, test conditions). Immediately export a plot of the Nyquist and Bode (magnitude/phase) plots for quick reference. Ensure that metadata (temperature, pressure, amplitude, SOC, operator, instrument, etc.) are noted either in the file or a separate log. Modern standards like the "Battery Passport" concept emphasize comprehensive data traceability over a cell's life 24 begin that at the lab stage by proper record-keeping.
- **Preliminary Analysis:** Identify the basic features of the spectrum: e.g., high-frequency intercept (ohmic resistance), any semicircles (charge-transfer or interfacial resistances with capacitive elements), Warburg tail (45° diffusion line at low freq if present), or inductive loops at high freq (often from leads or current collectors). Check that these features make sense (e.g. no negative resistances, which would indicate an error or oscillation). If something looks non-physical, consider re-measuring or troubleshooting the setup.

- Equivalent Circuit Fitting: If quantitative values are needed for specific elements (e.g. interfacial resistance or capacitance), fit the data to an equivalent circuit. Start with a simple model that reflects expected processes (for an interface-dominated impedance, a simple model might be: Rs (external series resistance) + [Rct || CPE] (charge transfer resistance in parallel with a constant-phase element for double-layer) + W_o (Warburg diffusion element) if diffusion is evident). Use the fewest elements necessary to achieve a reasonable fit 3. Caution: It is always possible to improve fit by adding extra RC elements, but these might just fit noise and have no physical relevance 3. Employ statistical criteria (e.g. chi-squared, residuals) and cross-validation (compare across multiple spectra) to avoid overfitting.
- **Result Reporting:** Document the interpreted results: e.g., "Interface resistance (R_{ct}) = 12.5 Ω at 25 °C, which is 10% higher than last batch's median (11.3 Ω)." Wherever possible, compare relative changes rather than absolute values e.g. an aging study might report that R_{ct} increased by 50% after a certain treatment, which is more robust than the absolute value alone. Use trends or percentile ranges if dealing with multiple cells.
- Traceability Links: Associate the EIS results with the specific cell or stack ID and lot number. In any database or report, tie in the fabrication parameters (for a solid-state battery, for instance: sintering batch, interface composition, etc.), test conditions (pressure applied, temperature, date, time, tester used), and even environmental logs (if in a glovebox, note dew point; if in ambient, note lab humidity). This ensures that months later, one can trace why a certain spectrum might look anomalous (perhaps humidity was high that day, affecting the solid electrolyte surface).
- Storage of Artifacts: Archive the raw data and analysis in the project's designated storage (e.g. a shared drive or data management system). Store images of the Nyquist/Bode plots (PNG/PDF) for quick access in presentations. If fits were done, save the fitted parameter values and equivalent circuit schematic used (perhaps in an Appendix or data repository). Consistent file naming and metadata embedding are key to future retrieval.
- Safety Final Check: After testing, de-energize any connections and return the cell to a safe storage state (especially if it was held at pressure or temperature—release pressure slowly to avoid jolting the cell; bring back to room temperature if heated). Log any incidents or abnormalities (e.g. if an alarm went off or if the cell exhibited a high self-discharge during the test). Always keep in mind that these guidelines complement, not replace, your organization's safety protocols and the instrument manufacturer's instructions.

1. Setup

The setup phase establishes the foundation for reliable and reproducible EIS measurements on the interface zone (IZ) of a battery stack. This section covers the physical configuration of the test (fixtures, connections, guarding), instrument calibration, and initial parameter selection, all in a **stack-agnostic** manner (i.e. applicable to various cell types and solid-electrolyte or liquid-electrolyte systems).

1.1. Test Fixture & Connections

A proper fixture and connection strategy is critical for minimizing measurement artifacts. **Use a four-terminal connection** (also known as 4-wire or Kelvin connection) to separate the current supply path from the voltage sensing path ²⁵ ²⁶. This eliminates the IR drop in lead wires from the measured impedance. Connect the two **current-carrying leads** to the opposite ends of the sample's series path, and connect the two **sense leads** as close as possible across the interface zone of interest ²⁵ ²⁷.

Arrange leads to mitigate electromagnetic interference: run the current leads as a twisted pair or use coaxial cables so their magnetic fields cancel, and physically separate them from the sense leads to

reduce mutual inductance coupling 5 6 . Mutual inductance between current and sense leads can induce an artificial inductive loop in the high-frequency impedance data, effectively appearing as a series inductance that isn't really in the cell 28 27 . By minimizing loop area (twisting leads carrying opposite currents) and maintaining distance between the current pair and sense pair, the coupling constant M (in the induced voltage V_s = M di/dt) is reduced, lessening error 29 30 . **Avoid unnecessarily high frequencies** if measuring a very low-impedance stack, because the induced voltage error grows with frequency (di/dt) 29 31 . For most cell interfaces, testing up to $^{\sim}100-200$ kHz is sufficient; going to MHz range may require extreme care or special cables due to inductive effects.

Shielding and guarding: Use a Faraday cage or at least a grounded metal plate around the test cell especially if the cell/stack has high impedance components (e.g. a solid-electrolyte stack at open-circuit). This protects against ambient noise (such as mains frequency interference). BioLogic notes that unshielded setups often pick up 50/60 Hz noise, which can overwhelm low-frequency data 32. An earthed Faraday cage will cut down noise dramatically, though it increases stray capacitance to ground by adding a nearby conductor 8. If the potentiostat/impedance analyzer has a **guard terminal**, connect the cage or shield to the guard rather than directly to ground. The guard is a driven zero-volt node that follows the potentiostat's reference, which can intercept leakage currents without affecting the measurement reading 9 33. In practice, many modern EIS instruments (e.g. Keysight, Solartron, Gamry) provide a guard connection that ties to the outer shield of cables. By guarding, any stray capacitance from the high potential lead to ground is effectively moved to between high lead and guard, so that the stray current does not enter the measuring amplifier 9 10 . **Practical tip:** Keep alligator clips, electrode pins, etc., clean and corrosion-free. High contact resistance at the clip interface can introduce a frequency-dependent contact impedance. As part of setup, verify continuity and low resistance of all leads by shorting them and measuring a milliohm-level impedance (this can be done using the instrument's short test function before connecting the cell).

Fixture selection: If testing stacked coin cells or solid-state pellets, use a fixture that can hold them securely with known pressure (more on pressure control in Section 4.2). The fixture material should ideally be insulating (or have guard shields) to avoid creating additional capacitances. For example, a simple 2-plate clamp can be modified by inserting a guard ring electrode around the sample area (driven at the same potential as the working electrode) to catch fringe currents. In multi-electrode configurations (e.g. a three-electrode cell with reference), ensure the reference is truly isolated and has a high input impedance electrometer to avoid loading errors ³⁴ ³⁵ (this is more relevant for half-cell studies; full-cell stack EIS often is two-electrode).

Grounding: Use a single-point ground to avoid ground loops. Typically, the impedance analyzer's ground (COM) should be the only ground point, and any metal enclosures or shields tied to that. Do not connect additional lab ground wires to the cell or fixture, as this can create circulating currents that disturb low-frequency measurements. If the instrument and any environmental chamber share mains power, ensure proper grounding through the outlet to avoid noise.

1.2. Calibration: Open/Short/Load Compensation

Even with an optimal fixture, the measured impedance includes contributions from the test leads and fixture itself (especially at the frequency extremes). To get accurate, "sample-only" data, perform **open circuit** and **short circuit** compensations (also known as fixture calibration). According to Keysight's impedance measurement guidelines, open/short compensation mathematically subtracts the parasitic

admittance of the open fixture and the residual impedance of the shorted fixture from the DUT measurement 36 37 .

- Open Compensation: With no sample in place (fixture open), measure the stray capacitance and conductance of the setup. Keep the fixture in the exact configuration used for the real measurement (same electrode spacing, same cables arrangement) to capture the true stray capacitance ¹¹. The open measurement ideally yields a very small admittance (since open circuit should pass ~0 current), but in reality, you'll see a capacitive admittance increasing with frequency. The instrument stores this open baseline: any admittance measured during the actual run that corresponds to this stray is subtracted out ³⁶. **Precaution:** Ensure that during open calibration, nothing is touching the electrodes (e.g., no accidental slight conduction) and the environment is similar (same shielding, etc.), because any difference can lead to under- or overcompensation. Keysight notes that performing open calibration with different electrode spacing or environment will lead to error ¹¹.
- Short Compensation: Short the working and counter electrode connections together (for a two-electrode test) or use a shorting bar/device across the terminals of the fixture. This measures the residual series impedance of the leads and contact points. Ideally the short should be as direct as possible; a poorly conducting short or one with significant length (introducing inductance) can itself introduce error 13 . The measured inductive reactance (and small resistance) of the shorted configuration will be stored during sample measurement, this is subtracted, removing most of the lead inductance effect. **Precaution:** Use the same tightening or contact pressure for the short as when the sample is inserted, to mimic the contact resistance. Also, if measuring over a broad frequency range, note that a shorting device may not be ideal at all frequencies (e.g., a shorting plate could have 20 nH inductance which is negligible at low freq but not at high). Some protocols use both open/short and an additional **load compensation**: measuring a known load (like a precision $100~\Omega$ resistor) to fine-tune the phase offset. This is more common in network analyzer-based EIS, but if your system supports it, it can improve accuracy further, especially in midfrequency range where neither stray capacitance nor lead inductance dominates entirely.

After compensation, verify by measuring a known component (for example, a dummy circuit or a resistor) to ensure the magnitude and phase come out as expected (the resistor should show purely real impedance at all frequencies, etc.). Proper open/short compensation will greatly flatten the high-frequency end (no spurious inductive upturn) and eliminate a low-frequency offset (no excessive tail from residual leakage). It essentially corrects the measured impedance Z_{xm} to the DUT impedance Z_{dut} via: Z_{dut} = Z_{xm} - Z_s - (Z_{xm} - (Z<sub>xm</sub

Guarding vs. calibration: Note that guarding (Section 1.1) and open/short compensation address similar error sources (strays) in different ways – guarding prevents stray currents from entering the measurement, while compensation subtracts their effect after measuring them. It's best practice to implement both if possible: physically guard and then run open/short calibration for the cleanest baseline ³⁸ ³⁹.

1.3. Instrument Settings (Amplitude & Frequency)

Selecting appropriate measurement settings for amplitude and frequency is a balancing act between data quality and physical validity of the impedance data.

AC amplitude (voltage or current): Start with a small AC amplitude to ensure the cell's response is in the linear regime. A perturbation of ~ 5 mV (rms) is a common default for potentiostatic EIS on batteries, and generally one does not exceed 10 mV 14 . Within this range, most electrochemical systems exhibit pseudo-linearity – the current response is approximately proportional to the voltage and primarily at the fundamental frequency, not generating significant harmonics 1 2 . This satisfies the linearity requirement for EIS theory. If using galvanostatic EIS (less common for interface studies, but possibly used for very low-impedance cells or when controlling current), choose an AC current amplitude that causes a similar small voltage oscillation (e.g. target 5 mV perturbation = i * |Z| at mid-frequency).

Non-linearity check: The amplitude should be low enough that the system's I–V curve is probed only in a near-linear segment. One way to verify this is by monitoring for harmonic distortion – many modern systems display a Lissajous figure or even calculate total harmonic distortion (THD) of the response. A clean sine wave current vs voltage Lissajous (an ellipse) indicates linearity; any distortion indicates the amplitude might be too high. Gamry Instruments emphasizes that using the default 5–10 mV is usually safe, but there are cases where a higher amplitude is needed (e.g. very low impedance cells might require several mA or a larger perturbation to get above noise) ¹⁵ . If you must increase amplitude (due to noise concerns), do so gradually and watch for signs of non-linear response (e.g. the appearance of a "loop" in the Nyquist at low frequencies that might actually be a time-domain distortion artifact). According to Gamry, too small an AC signal yields noise-prone data (high scatter, especially at high frequency for low-Z cells or at low frequency for high-Z cells), whereas too large yields non-linear data (falsified results) ¹⁵ ⁴⁰ . There is no universal perfect amplitude; it depends on the cell. For high-power cells, an amplitude of a few hundred microamps or millivolts might be too small to measure; for a delicate interface (like a passivated lithium surface), even 10 mV might be too large if it breaks down a film.

Frequency range: Cover a frequency spectrum broad enough to encompass all relevant electrochemical processes of the interface zone:

- The **high-frequency limit** is often set by the equipment (many lab potentiostats go up to 100 kHz or 1 MHz). For battery interfaces, frequencies above a few hundred kHz primarily capture inductive behavior from leads or perhaps contact resistances and very fast dielectric processes. If your instrument and fixture are well-calibrated, you can include data up to ~1 MHz; just be cautious interpreting any feature there as it could be instrument/fixture-related. Some standards use a single frequency point of 1 kHz for internal resistance measurement ⁴ (for example, EV battery production lines often test at 1 kHz AC ⁴), but full EIS often extends higher to see the onset of inductance.
- The **low-frequency limit** should be as low as needed to capture the slowest process of interest (e.g. diffusion through a solid electrolyte or across an interface layer). For lithium-ion cells, 10 mHz (0.01 Hz) is a common lower limit, requiring ~100 s per cycle such a sweep can already take an hour or more for enough points. Solid-state batteries with blocking interfaces might need even lower frequency to see complete Warburg diffusion tails, but going too low risks the system drifting during measurement (thus violating steady-state). It's often a trade-off: in research, 1 mHz is sometimes attempted, but in a production or QA environment, one might stop at 100 mHz or 10 mHz to save time and reduce drift. **Tip:** If low-frequency data is crucial but time-consuming, consider measuring a few strategic low frequencies with longer wait (e.g. hold 1 mHz until the

response stabilizes) rather than a dense sweep, or use techniques like Pseudo-Random Binary Sequence (PRBS) EIS or Fourier methods to accelerate (advanced topic, not covered here).

Frequency distribution: Use a logarithmic sweep (constant number of points per decade). Ensure enough points (at least ~5 or more) per decade to define the shape of semicircles or Warburg lines accurately. Some modern instruments allow adaptive frequency steps – those can be useful (they take more points where the impedance is changing rapidly). If you have prior data or expectations (e.g. you know there's a semicircle around 1 Hz), you might densify around that. Typically, one might do ~50 points from 100 kHz to 0.1 Hz as a starting plan. Remember, each decade lower doubles the measurement time (approx) if using single-sine method.

Integration time and averaging: For each frequency point, the instrument applies a sine wave for a certain number of cycles to get a stable reading. Many systems default to 1–3 cycles of settling then a few cycles of data averaged. Increase the integration time at low frequencies to get a stable reading (the first cycle might be influenced by transients). Also consider repeating the lowest frequency point to check if it was stable (some software automatically do a second pass or a loop until two readings agree within tolerance).

Initial test run: It's advisable to do a quick trial run on a sample (or even a shortened frequency range) to see if the amplitude is appropriate and the data quality is good. This trial can reveal if adjustments are needed (e.g., if you see a strange spike at some frequency, you might reduce amplitude or check shielding at that frequency).

Table 1 in **Appendix A** summarizes recommended starting values for key EIS parameters (amplitude, frequency range, etc.), along with rationale and pitfalls.

1.4. Environmental Controls

Stability of environmental conditions during EIS is paramount, because the interface impedance can be highly sensitive to temperature, pressure, and other factors. The goal is to **maintain constant temperature and pressure** throughout the measurement and ensure the environment does not introduce time-varying disturbances (like drying out or humidity ingress).

Temperature: Conduct measurements in a controlled temperature environment. For R&D, this could be a temperature chamber or a climate-controlled lab glovebox. Temperature affects reaction kinetics (and thus charge-transfer resistance) and ionic conductivity (thus bulk resistance) exponentially (Arrhenius behavior) 41 42. Even a few °C drift can change the impedance noticeably. Set the temperature setpoint (e.g. 25.0 °C) well before the test and allow the cell to equilibrate. If using a chamber, be aware of thermal gradients – ideally, place a thermocouple near the cell to verify actual sample temperature. If measuring multiple cells in sequence, keep them in the chamber for consistency. **Note:** If the test is performed in a glovebox for moisture control (common for solid-state cells), the glovebox temperature might be slightly above ambient due to circulating fans. Monitor it and record it. Also, the **dew point** in the glovebox is important: a sudden rise (glovebox leak or an event) could introduce moisture that alters a solid electrolyte interface impedance drastically. Log the humidity or dew point during the test if applicable. Public literature on fuel cells and membranes has shown EIS is used to detect water content ⁴³ – similarly, in solid-state batteries moisture can produce spurious conductivity paths. Thus, environment logging (humidity, etc.) is part of good practice.

Pressure: Many interface-zone studies, especially for solid-state batteries (SSB), involve applying a **stack pressure** to ensure good interfacial contact. The impedance can change with pressure: higher pressure

often decreases contact resistance up to a point (by increasing the true contact area or reducing microgaps). However, pressure can also affect the materials (e.g. deforming layers). Therefore, whether you choose a nominal pressure of 1 MPa, 5 MPa, 20 MPa etc., keep it consistent for all comparative tests. Use a calibrated mechanism (spring gauge, hydraulic press with gauge, or a screw with known torque correlated to pressure on a given cell area). As an example, a research study on pouch cells compressed between 100 kg and 1000 kg force (approx 0.3 to 2.7 MPa on their cell area) showed that increasing pressure significantly altered the low-frequency impedance (increased R_{ct} at low SOC) 44 – a counterintuitive result attributed to pressure-induced electrode changes. This underscores that any reported impedance must be tied to the pressure condition. **Control:** If possible, apply the pressure and then wait (as noted in Section 1.3) for any relaxation. Some fixtures even allow actively maintaining pressure as the cell relaxes (springs). Avoid doing EIS while the pressure is still changing (tightening screws during measurement is a big no). Record the pressure value and how it was applied (since friction in a screw thread could make torque an imprecise measure, for instance).

Vibration and External Noise: Environmental vibrations (from nearby equipment, building HVAC, etc.) can introduce noise in the measurement electronics or physically perturb the cell (which might modulate pressure or contact). While EIS is generally low-amplitude and not highly susceptible to vibration, extremely low-frequency measurements could be influenced if, say, something causes periodic pressure oscillations. If you suspect such issues, isolate the setup on a vibration-damping table. Electrically, keep AC mains-powered devices away from the measurement cables; use shortest possible cables. It's good to perform a "cell open" measurement with everything connected (but cell not installed) to see if you pick up any environmental noise – a flat line near zero on the Nyquist indicates a quiet environment, whereas any stray points (especially at specific frequencies like 50 Hz, 60 Hz, or their harmonics) indicate noise sources that should be addressed (shielding or turning off sources).

With the setup properly configured – fixture in place, instrument calibrated, parameters chosen, and environment steady – we move to the execution phase, where the actual EIS measurement is carried out on the cell/stack.

2. Execution

Execution covers the steps from just before starting the impedance measurement through the end of data acquisition. At this stage, the cell is in place, the equipment is configured, and we ensure the procedure runs smoothly and yields valid data. Key aspects include verifying steady-state (OCV) before start, controlling the test sequence, and monitoring for any anomalies during the run.

2.1. OCV Stabilization & Pre-test Conditioning

Before applying any AC signal, confirm the cell or interface is at a stable operating point (usually open-circuit, no net DC current). This satisfies the EIS requirement of time-invariance during the test. As mentioned earlier, wait until the **Open-Circuit Voltage (OCV) is stable**. For instance, if you assembled a Li metal|solid electrolyte|Li metal symmetric cell to probe the interface, you should wait after cell assembly for the OCV to reach essentially 0 V and current to die down (indicating minimal net reaction). If measuring a full cell at some state-of-charge, ensure it's been resting sufficiently after the last charge/discharge step – typically, at least 2–3 hours rest is recommended for Li-ion cells to reach equilibrium potentials (some slower processes can take longer, but practicality wins). Any drift in OCV during EIS will appear as a spurious element in the impedance (because the system is then slightly non-linear or time-varying).

One way to test for stability is to do a quick low-frequency scan manually: apply a small DC step (maybe a few mV) and see if a steady current holds or decays. However, usually simply monitoring the voltage is enough. For OCV cells, the criterion could be, for example, <0.1% change over the entire EIS test duration expected. Gamry's EIS basics guide points out that measuring an EIS spectrum often takes many minutes to hours, and "the system being measured must be at a steady state throughout [that] time" $\,^{17}$. They warn that common causes of error are slow drifts such as adsorption or oxide growth changing the system during the run $\,^{45}$. If such drift occurs, the EIS data might fail KK validation or fit oddly.

Electrochemical conditioning: In some cases, especially with fresh interfaces (e.g., a freshly made lithium/electrolyte interface or a freshly assembled cell), a small "conditioning" step is applied prior to EIS. This could be a brief DC polarization to stabilize a film or a formation cycle on a battery to establish the SEI. Make sure any conditioning is done well before EIS and that any transients from it have decayed. For example, after a formation cycle, let the cell rest at OCV for a specified duration (some protocols call for 24 hours rest for full cells to stabilize the SEI before characterization).

Equilibration under test conditions: Ensure the cell is at the test temperature and pressure for some time before starting. If you moved the cell into a chamber at 40 °C, give it time to uniformly reach 40 °C internally. If you just applied a 5 MPa pressure, the cell stack may slowly deform over tens of minutes; let the initial creep happen before EIS. The IEST study earlier explicitly waited 20 min after each pressure increment before taking EIS readings ¹⁸, which is a good guideline.

Finally, document the starting OCV, temperature, pressure, and time. Start a fresh entry in the lab notebook or digital log for this run, noting "Cell ABC, OCV = X.XXX V, T = 30.0 °C, P = 5 MPa, starting EIS at time HH:MM."

2.2. Pressure and Temperature Control

During the measurement, maintain the **pressure and temperature constant**. If using a temperature chamber, do not open the door or otherwise disturb it until the test is finished (opening could cause a temperature drop and also electrical noise). If using a pressure device like a screw press, ensure it doesn't relax; devices with springs or pneumatic regulators are better for maintaining constant force as the cell may slightly change thickness. Log any readings of pressure if your fixture provides them (some advanced fixtures have a load cell to monitor live pressure – if so, that data could even be recorded alongside impedance in some setups).

If at any point you observe a rapid change (say the chamber overshoots temperature or an unexpected pressure drop), you might consider aborting and restarting once stable. It's better to have a clean dataset than to push through a perturbation and get compromised data.

For long tests, temperature controllers with good stability (± 0.1 °C) are ideal. If your lab has significant day/night swings and the test spans that, be cautious – maybe schedule impedance tests in a period where the environment is most stable (some labs do EIS overnight when there's less human activity causing temperature or electrical noise changes).

2.3. Measurement Procedure & Monitoring

With everything ready, initiate the EIS frequency sweep (or multi-sine, or whatever method chosen). Here's what to do during the run:

- High to low frequency sweep: Most systems default to start at the highest frequency and go down. This is generally preferred because high-frequency data comes quickly (within seconds) and gives an immediate sense if the cell connection is good (you see the bulk resistance right away) and if the open/short comp was done (you might see a near-zero imaginary part at high freq if done correctly). It also means the longest measurements (low freq) happen last. If the cell were to drift, that drift mostly affects the tail end one can decide to truncate if needed. In contrast, a low-to-high sweep could run into drift early and ruin the rest. So stick with high→low.
- Live monitoring: If your instrument software shows the Nyquist plot populating in real time, watch it. Check that the points look reasonable: e.g. they should typically start near the real axis (ohmic resistance) at high f and then may go into a semicircle or line. If the very first point is wildly off (e.g. negative Re(Z) or huge), that could indicate a problem (like cables not properly zeroed). Sometimes the first point at max frequency is less reliable if the instrument didn't fully sync; a slight anomaly at the very first point can sometimes be ignored or measured again later. But large anomalies anywhere should prompt you to consider pausing. Many systems allow pausing or even aborting and then continuing (though continuing after abort might risk losing data continuity).
- Compliance and saturation: Ensure the potentiostat isn't hitting its compliance limits. For example, if measuring a very high-impedance interface (hundreds of $k\Omega$), the small AC current may be at the limits of the electrometer to sense. If you see the phase becoming erratic or the magnitude extremely noisy at high frequencies for a high-Z sample, it could be that the current is below the resolution. In such a case, you might need a higher amplitude or a different approach (like two-electrode vs three-electrode setup to reduce uncompensated resistance). Conversely, for low-impedance cells, ensure the current being drawn by the AC perturbation is within the potentiostat's capability. A 5 mV amplitude on a 1 m Ω cell means 5 A AC current (!), which most lab instruments cannot do. In such cases, one must switch to galvanostatic mode or limit the frequency range (because at high freq the impedance might approach that low value). Hioki's battery tester notes that very high-capacity cells have extremely low internal resistance, making it hard to measure accurately with standard 1 kHz methods ⁴⁶ ⁴⁷ specialized instruments or techniques are needed to measure in the $\mu\Omega$ range. If you're in that regime, double-check that your instrument can handle it, or measure a slightly higher impedance by perhaps limiting the cell size (maybe test a single layer instead of a full module).
- Interruption handling: Ideally, avoid any interrupts during the sweep. Don't start other high-power equipment on the same circuit (to prevent voltage spikes). Don't use the computer controlling the test for other tasks that could cause USB or CPU load issues. If the software allows adding markers or notes, mark down if you did notice anything (e.g. "point 35Hz looked noisy, possibly someone walked into lab with phone ringing" EMI events can happen).
- **Completion:** Let the sweep complete fully to the final frequency. Once done, many systems do some calculations or automatically run a KK test or error check allow those to finish.
- **Duplicate point check:** As a quick sanity check at the end, you can manually re-run one frequency to see if the result is same. For example, after completing, apply a single-sine at 1 kHz and see if the impedance matches the earlier 1 kHz point. This can reassure that nothing

fundamental changed. Some protocols actually run a few frequencies at the beginning and end for this purpose.

At the end of execution, you have an impedance spectrum dataset. The next steps involve quality assurance and validation of this data before trusting it for interpretation.

3. QA & Consistency Checks

Quality assurance (QA) for impedance data ensures that the obtained spectrum is credible, free from major experimental artifacts, and repeatable. In this section, we cover using Kramers–Kronig tests for internal consistency, doing repeat measurements or statistical analysis for precision, and establishing criteria to accept or reject data sets. We'll also discuss recognizing common signs of bad data.

3.1. Kramers-Kronig Validation

The **Kramers–Kronig (KK) relations** are mathematical constraints that any physically valid impedance data must satisfy, stemming from the principles of linearity, causality, and stability of the system. In essence, the real and imaginary parts of the impedance are Hilbert transforms of each other for a passive stable system; they are interdependent. Therefore, a KK consistency test takes the experimental $Z(\omega)$ data and attempts to reconstruct either Re(Z) from Im(Z) or vice versa, checking for discrepancies 20 .

A well-behaved impedance spectrum will pass the KK test with only minor deviation within experimental noise. A failed KK test (large deviation) indicates one or more of: - Violations of EIS assumptions: the system was not perfectly linear (maybe due to too large an amplitude or a drifting OCV) or not time-invariant (e.g. changing during the test), or not causal (this last one is usually not an issue unless reference electrode problems or such). - Insufficient frequency range: If you didn't measure low enough in frequency, the KK transform trying to predict the 0 Hz behavior might be off. Likewise for the high end. - Data noise and artifacts: random noise can cause issues, or if you have points that are outliers, KK will treat them as real and then the reconstruction doesn't align well.

Performing KK test: Many software packages (Bio-Logic EC-Lab, Gamry Echem Analyst, Zahner, Autolab, etc.) have a KK validation tool. Some fit the data to a KK-compliant model (Voigt or Foster circuits) and report a pseudo chi-squared error ⁴⁸. Others directly integrate the KK relations. The output might be a plot of measured vs KK-predicted impedance or simply an error metric. For instance, one method is to calculate a "consistency" parameter – e.g., mean absolute percentage error between original and KK-fit data. A result under 5% is often considered good; >10% is concerning.

Handling failures: If your data fails KK, do not immediately discard it; first consider if the failure is explainable. Example: You measured from 1 Hz to 1 kHz only – that limited band might inherently not satisfy KK perfectly because the true system has dynamics outside that band. In Bio-Logic's application note #15, they show a truncated spectrum (missing low frequencies) clearly doesn't match a KK reconstruction until they fit it with an assumed circuit to extrapolate ⁴⁹ ²³. They solved it by fitting to a Voigt circuit (a series of RC elements) which is KK-valid by construction, demonstrating the truncated data could be representative ⁵⁰ ⁵¹. Thus, sometimes you can "force" consistency by modeling. However, if you have the ability, it's better to extend the measurement range or improve conditions and remeasure.

If KK indicates a problem and you suspect drift: check the OCV before vs after as earlier noted. If there's a difference, likely the KK failure is due to that drift. The data might show a "loop" or negative-resistance artifact at low frequency in such cases.

Automated KK in instruments: Some modern instruments (e.g. Solartron Modulab) can do a KK check on the fly or immediately after a run. Use that if available, as it may prompt re-testing quickly.

In summary, use KK as a **gatekeeper** for data quality: don't over-interpret an EIS spectrum that doesn't pass basic consistency checks. It's better to repeat the test under improved conditions.

3.2. Repeatability and Drift Checks

A single EIS run provides one instance of data. Good practice is to assess **repeatability** – the variation when the same test is repeated under identical conditions – and ensure it's within acceptable bounds.

Intra-run repeatability: As mentioned, sometimes repeating immediately can reveal if the first run altered the system (for example, if the first EIS partially conditioned the interface). If the second run is significantly different, the system may not have been stable to begin with, or the measurement itself induced change (usually EIS is non-destructive, but at too high amplitude or on very fragile interfaces, it could perturb them).

Inter-run variability: If you have multiple nominally identical samples (say several cells from the same lot), measuring all and looking at the spread in impedance features gives an idea of natural variability and measurement error combined. For QA in manufacturing, one might define control limits: e.g., interface resistance must be 50 Ω \pm 5 Ω for acceptance. Statistical approaches like averaging multiple spectra or computing standard deviation at each frequency can highlight frequency ranges with more noise (often high freq phase and low freq magnitude have larger variance).

Drift during measurement: We touched on this – check for drift by comparing initial vs final quick measurements. Another method is splitting the spectrum: measure low-to-high then high-to-low back in one sequence and see if they overlap. If not, drift occurred during the first pass. One can also include a duplicate frequency point halfway through the run to see if it changed (some people repeat a mid-band frequency periodically).

Recognizing bad data patterns: - If you see a tiny tail curling back at low frequency forming a small loop (sometimes even a negative Im(Z) at very low freq in a Nyquist), that often indicates an artifact (inductive loop from leads or a slight oscillation). Real electrochemical systems can have inductive loops if there's a relaxation process, but in batteries it's uncommon except maybe at very high SOC or weird conditions. - Scatter (noisy zigzag) in the data, particularly in the Bode phase plot, usually means the signal-to-noise was low at those points. If it's just a bit of scatter but overall shape is discernible, you might smooth or ignore minor points. But if whole sections are noisy, you may need to re-run with higher amplitude or longer integration. - A semicircle that isn't a nice round shape but has a sharp cusp could mean something like instrument range switching happened. Some instruments switch gain at a certain impedance threshold and sometimes that transition shows. One way around that is to manually set ranges or ensure proper open circuit calibration so range switching is seamless.

Batch QA: In a pilot or production context, you might measure EIS on every cell in a batch. It's useful to automate QA flags: for instance, automatically flag any cell whose high-frequency resistance is out of expected range (like an outlier, which could mean a bad weld or contact in that cell), or whose mid-frequency arc is too large (perhaps indicating a high interface impedance that could correlate with a defect). Over many cells, you can establish baseline distributions for each parameter.

Table 2 in **Appendix B** outlines some QA steps (KK test, repeat measurement tolerances, etc.), the methods to perform them, and example pass/fail criteria.

3.3. Data Acceptance Criteria

Before proceeding to interpretation, define what constitutes "acceptable" data for your purpose: - **KK consistency:** must be within X% (as above). - **Repeatability:** a repeated measurement of the same sample should vary by no more than Y% in critical parameters (perhaps ~5% for resistances is a reasonable goal in many lab setups; capacitances often vary more). - **No unphysical values:** e.g., negative resistances or capacitances, wildly erratic phase > $\pm 90^{\circ}$ for simple systems, etc. Some fitted parameters like CPE exponent (n) should be in a physically plausible range ($0 \le n \le 1$); if your fit yields n = 1.2, something's off (fits can do that if data was bad). - **R(test) vs R(reference):** If you have a reference sample or previous data, ensure the trend makes sense. For instance, if all previous similar cells had ~10 Ω interface resistance and today you got 0.1 Ω or 100 Ω , that's likely wrong – investigate before accepting.

If data fails criteria, the action is usually to retest after fixing any suspected issue. It's much cheaper in time to repeat an EIS than to waste effort analyzing bad data or, worse, making false conclusions.

Documentation of QA: It's good to include in the report of that EIS (especially for formal reports or if sending to a customer) a note like "KK test passed with <2% error; duplicate measurement within 3% on key metrics – data considered reliable." This builds confidence in your results.

4. Data Interpretation

After obtaining high-quality impedance data that passes QA, the focus shifts to interpreting what the data means for the interface zone stability and properties. This involves modeling the data with equivalent circuits, extracting parameters, comparing across samples or conditions, and drawing conclusions about the interface (e.g. has it aged? does one processing lead to lower resistance? etc.). Key points here are to use appropriate equivalent circuit elements for the interface, avoid overfitting or over-interpreting, and emphasize comparative analysis for decision-making.

4.1. Equivalent Circuit Modeling

Equivalent circuits are used to quantify different processes represented in an EIS spectrum. Each element in the circuit corresponds (ideally) to a physicochemical process: - R (resistor): often represents a resistive process like ionic conduction through a electrolyte, electronic conduction through an electrode, or a contact resistance. For an interface zone, we often consider a series resistance R_b (bulk resistance, e.g. of electrolyte or electrode) and an interfacial resistance R_int (e.g. charge transfer resistance at an electrode/electrolyte interface or grain boundary resistance in a ceramic electrolyte). - C (capacitor): represents a pure capacitive behavior, such as a dielectric or double-layer capacitance at an interface. However, pure capacitors are rarely seen; non-ideal behavior leads to use of CPEs. - CPE (Constant Phase Element): a component with impedance $Z_{CPE} = [Q(j\omega)^n]^{-1}$ that generalizes a capacitor (if n=1, Q=C exactly; if n<1, it's a depressed semicircle). CPEs empirically model the distributed capacitance or surface inhomogeneity at interfaces. They are very commonly used in battery impedance fits because real interfaces rarely show ideal capacitive arcs - instead, you get depressed semicircles (often $0.8 \le n \le 1$). - Warburg impedance (W): represents a diffusion process. There are different types (finite length Warburg with open or short termination, infinite Warburg). A Warburg manifests as a ~45° line in Nyquist if semi-infinite diffusion, or as a spike that transitions to a second semicircle if finite-length diffusion. For solid interfaces, a Warburg might model diffusion of Li through a solid electrode or across an interphase layer. - Inductor (L): usually accounts for inductive behavior at high frequency from leads or coils. If after open/short calibration you still see a bit of inductance (initial upward slope), you might include a small inductance L in series with the whole circuit. - Gerischer, etc.: There are more exotic

elements (Gerischer impedance for certain reactions, etc.), but these are less common and beyond our scope.

When modeling the interface zone of a battery stack, consider a **physical picture**: For example, in a lithium metal solid-state battery, the interface impedance might include: a contact resistance (R_contact) in series, a charge transfer or interface reaction resistance (R_ct) in parallel with a double layer capacitance (C_dl or CPE), and possibly a Warburg for diffusion of Li through the interface or into the electrode. So an equivalent circuit guess might be: R_ser (series) [R_ct || CPE_dl] (series) W. For a symmetric cell (Li|SE|Li), you might have two identical interfaces in series, which effectively halves the arc (two arcs of same size merge into one half-sized arc if truly identical and measured in series), but often one just models as one interface and acknowledges it's two in series.

Fitting process: Start with the simplest circuit that qualitatively matches the data. For instance, if you see one depressed semicircle and a Warburg tail, use $R_{ser} + [R||CPE] + W$. If you see two semicircles, you might have $R_{ser} + [R1||CPE1] + [R2||CPE2]$ etc. Each added element should be justifiable (maybe one semicircle is the high-frequency SEI, another is the charge transfer). Remember Ockham's razor: more parameters can always fit better but may not be unique or meaningful 3 . There is often not a unique equivalent circuit for a given spectrum 52 ; multiple circuits can produce similar impedance, so use knowledge of the system to choose.

Physical constraints: Impose constraints based on physics: e.g., capacitances should be in a realistic range (double layer capacitances $\sim \mu F/cm^2$ order of magnitude, so if you have an interface of 1 cm², expect maybe tens of μF , though CPEs complicate direct interpretation; a very large fitted Q might indicate it's not a simple double layer but some battery blocking capacitance). Resistances should be positive and roughly consistent with DC measurements (if you measure the cell's DC resistance via a pulse, it should correlate with the sum of certain impedance components like R_ser + R_ct at low freq intercept).

Interpreting fitted values: Once you get a fit that matches the data (low residuals, random residual pattern), interpret the values in context: - R_ser (often noted as R_s or R_ Ω) is typically the high-frequency intercept on the real axis of Nyquist. It corresponds to the Ohmic resistance: contributions from electrolyte, current collectors, etc., basically everything that is not frequency-dependent in that range. For solid electrolytes, this might be the bulk ionic resistance plus maybe grain boundary if that is very fast. - R_ct (charge transfer resistance) is related to the kinetics of the interfacial reaction (like Li* crossing the interface). If this value increases, it could mean a less active interface (aging, increased interphase layer). - C_dl or CPE parameters: these might give an idea of interface area or dielectric properties. Often, one might convert a CPE to an "effective capacitance" at a particular frequency or use Brug's formula if needed to compare to expected double-layer capacitance. - Warburg coefficient (σ): if you have a Warburg element, you can extract diffusion coefficients if you know the relationship (for planar diffusion: $Z = \sigma(1-j)\omega^{-1/2}$, and σ relates to D via $\sigma = \frac{RT}{AF^2C\sqrt{2D}}$ for certain cases). This might be too deep for routine QA, but mention it if relevant to understanding (e.g. diffusion through an interphase got slower, maybe the interphase thickened).

Example interpretation: Suppose after fitting, you find R_ser = 5 Ω , R_int = 20 Ω , CPE-Q = $5 \times 10^{\circ}-5 \, \text{S} \cdot \text{s}^{\circ}$ n, n=0.9. If the previous generation interface had R_int = $10 \, \Omega$ under same conditions, now it's doubled – a sign of poorer kinetics, perhaps due to a new interlayer. The CPE-Q maybe corresponds to an effective capacitance of say $50 \, \mu\text{F}$ (just an example), which might align with an area of a few cm² of double-layer, etc. These interpretations help link back to physical changes: e.g. maybe the interface formed a resistive layer (R_int up) but still has similar capacitance (so likely the same area, just more resistive).

Don't over-trust absolute fits: It's worth reiterating that equivalent circuit parameters can sometimes trade off (e.g. a slightly different C with different n can fit similarly). Therefore, use them as comparative indicators. If all samples are fit with the same model, comparing R_c t across them is usually robust even if each one had, say, $\pm 10\%$ uncertainty, the trend is meaningful.

4.2. Avoiding Overfitting & Comparative Analysis

Avoiding overfitting: As we have stressed, adding more elements can always reduce the fitting error, but at the cost of interpretability and sometimes stability of the fit. An overfit model might assign a tiny RC pair to some noise wiggle. Gamry's guide explicitly warns: "You can always get a good looking fit by adding additional circuit elements... these elements may have little relevance to the cell processes under study" 3. To avoid this: - Limit initial fits to the number of features you clearly see. If there's one arc and a tail, do one RC + Warburg, not two. - If the fit is not perfect, consider whether the deviation is systematic (maybe your model is missing an element) or random (noise, which you shouldn't fit). - Use cross-validation: For example, fit two datasets from similar cells with the same model and see if parameters are consistent. If one dataset demands an extra element that the other doesn't, maybe that extra element was just noise in one. - Physically justify each element: If you add a second CPE-R pair, what could it be? Perhaps an SEI film vs charge transfer; if that's plausible, fine. If you're adding a third just to reduce error, question it.

Comparative analysis: Often the goal in QA or research is not to determine an absolute value with ultra precision, but to see differences: e.g., does a new coating reduce the interface resistance? Is cell A's interface more stable over cycles than cell B's? For such questions, you may not even need to do a full equivalent circuit fit for each – sometimes simpler metrics suffice: - The high-frequency intercept (ohmic resistance) can be read directly from the plot. - The diameter of the semicircle (difference between high and low frequency intercept of that arc) gives the charge transfer resistance approximately. - The slope of the low-frequency tail can indicate diffusion impedance. - These can be compared sample to sample directly on the plots (overlaying Nyquist plots is very illustrative).

It's often useful to include a **percent change** or **ratio**: e.g., "After 50 cycles, the interface resistance increased by 200% (from 20 Ω to 60 Ω)". Or "Cell with additive X had 30% lower low-frequency impedance magnitude than baseline." These are more meaningful for action than "it was 5 Ω vs 7 Ω " alone, especially given measurement uncertainties.

Use of percentiles and statistics: If you have many cells, you might report median values and ranges. For instance, "Baseline cells: R_int = $15 \pm 3 \Omega$ (median \pm IQR). New formulation cells: R_int = $8 \pm 1 \Omega$." This shows not just the average improvement but also consistency.

Graphical interpretation: Show the plots! A visual comparison can sometimes directly answer the question of stability: e.g., if the impedance spectrum doesn't change shape or size after a certain handling, that suggests stable interface. If a second semicircle grows over time, that suggests maybe a new layer forming. Encourage looking at Bode magnitude/phase too; sometimes what's a messy Nyquist can be clearer in Bode (phase peaks corresponding to time constants etc.).

Linking to other data: In a broader analysis, interface impedance from EIS could be correlated with other measurements like capacity fade, micrographs of interface, etc. For QA, you might correlate high interface impedance with cells that failed end-of-line tests. Keep an eye on such correlations as they validate the EIS's usefulness (e.g., if higher R_ct correlates with higher overpotential during formation cycling, that's expected).

In conclusion, interpret EIS results with a balanced approach: trust the trends and relative changes more than absolute exactness, use circuits as tools not truths, and always consider the context of how the interface behavior ties to performance and stability metrics.

5. Traceability & Data Management

Traceability is about ensuring that every EIS result can be traced back to the exact conditions and sample history, and that the data is stored in an organized way for future reference or audits. Given the complexity of battery manufacturing and R&D, establishing this traceability at the QA/EHS level is crucial. Here we outline how to document and link data, what metadata to capture, and how to store artifacts.

5.1. Metadata and Logging

For each impedance test, record comprehensive metadata. This typically includes: - Sample identification: Unique ID for the cell or stack. Include lot or batch number, and if relevant, the position in a module or any fabrication details that distinguish it. - Test conditions: Date and time of test, operator, location (lab station, glovebox #, etc.), temperature (setpoint and actual, e.g. 30.0 °C in chamber #5), pressure (e.g. 5 MPa using fixture XYZ), atmosphere (e.g. "in air, 45% RH" or "in Argon glovebox, dew point -50 °C"). - Instrumentation: Instrument model and serial (e.g. "Gamry Reference 600+ S/N 1234" or "BioLogic VSP-300 with 4 channels, channel 2 used"), software version, calibration status (if outside calibration due date, note it). - Measurement parameters: Amplitude (voltage or current) used, frequency range, number of points, mode (potentiostatic/galvanostatic), cell connection type (2electrode vs 3-electrode), reference electrode used (if any, and its type). - Cell state: OCV at start (and end), SOC or equivalent (e.g. "Cell at 50% SOC, 4.0 V vs 4.2 V max"), any recent cycling (e.g. "impedance measured after 3 formation cycles, cell rested 12h" or "after 100th cycle, 1h rest"), etc. Also note if the cell was fresh from assembly or aged. - Anomalies: Note anything unusual observed, e.g. "During test, noticed slight oscillation, possibly due to loose cable - resolved and repeated test" or "Chamber temperature fluctuated ± 2 °C due to compressor cycling". - Link to procedures: If there is an internal test procedure or SOP, reference it (like "Procedure EIS-01 followed").

Many labs use templates or software for this. For instance, some battery testing software or LIMS (Lab Information Management Systems) allow tagging EIS data with such metadata. Even a simple spreadsheet can serve as a log as long as it's diligently filled.

One concept emerging in industry is the **Battery Passport**, particularly in the EU, which envisions recording detailed data from production to end-of-life for each battery 53 54. While that covers more than EIS, it underscores a trend: data recorded now in R&D could feed into digital twins or databases that track how process conditions affect performance. Therefore, capturing rich metadata is not overkill; it's future-proofing.

5.2. Artifact Storage and Linking

Raw data files: Always save the raw impedance data file generated by the instrument. Ideally in a non-proprietary format (or alongside the proprietary format). For example, "Cell123_EIS_2025-09-19.DTA" (BioLogic) plus an exported CSV of frequency, Z', Z", etc. These files should reside in a structured file system or database. A hierarchy might be: /Project/ExperimentDate/Cell123/EIS/... etc. Consistency is key.

Processed data: If you perform analysis like fitting, save those results too. This might be a screenshot of the fit, or an exported table of fit parameters. If using a tool like ZView or EC-Lab to fit, you can often save

a project file with the circuit and results. Save those with versioning (if you re-fit later with a different model, keep the old one too but note which is considered final).

Plots: Save image files of the impedance plots (at least Nyquist, maybe also Bode). Annotate them if helpful (e.g. mark which arc is which, or add labels). These can be embedded in reports or presentations. Ensure the image is clear and includes a legend if comparing multiple cells.

Linking data to other results: Often EIS is one of several tests. For full traceability, link it to, say, the cycle life data of that cell, or the microscopy of that interface. Practically, this could mean naming files with the cell ID that can be cross-referenced, or having a master table: Cell123 – EIS filename – formation capacity – microscopic imaging filename, etc. The goal is that someone could later pick up the EIS result and quickly find all related info about that cell.

Database usage: If available, use a database to store EIS results. There are efforts in the community for standardized data formats (for example, **ECMD** – Electrocatalysis Modelling Database format, or others for battery data). One recent work proposed a hierarchical data format for battery testing including EIS, to allow easier data sharing and machine readability ⁵⁵ ⁵⁶. In absence of a specialized database, even a well-structured set of folders with an index file can do.

Backup and archives: Because EIS data can be quite valuable for long-term understanding (you might measure today and not fully analyze it until after several months of other experiments), ensure data is backed up. Follow your organization's data retention policies – e.g., maybe raw data is kept on a local PC for a month but then moved to a server or cloud. Verify that the data can be opened later (especially proprietary formats – be cautious that software updates or license availability might hinder opening old files; exporting to CSV ensures you have the numbers regardless).

Documentation for QA/EHS: From an EHS perspective, also document any safety or quality issues observed during testing. For example, if a cell vented or got hot (which can rarely happen even during an impedance test if a short occurs), log that incident and trace it to the cell's impedance data. This could help in root cause analysis.

All these traceability measures ensure that the impedance data is not an isolated piece of information but part of the larger puzzle of manufacturing quality. It allows trends to be spotted (e.g., all cells from Lot42 have slightly higher interface impedance – maybe something changed in that lot's process), and it allows accountability (you can answer "when, how, and under what conditions was this data taken?" with confidence).

6. Safety and Compliance Considerations

While EIS is generally a low-risk, non-destructive test, working with batteries and electronic instruments still entails safety considerations. This section highlights safety and compliance points to keep in mind, especially as they relate to publicly available guidelines or standards. Remember that **site-level SOPs and safety rules override any generic advice here** – always follow your organization's protocols first and foremost.

Electrical safety: Use properly rated cables and fixtures for the voltages and currents involved. Most EIS on single cells is low voltage (a few volts) and low current (mA), but if you're doing stack modules or higher-power EIS (some advanced EIS can be done on battery packs), ensure the instrument is designed for it. Many potentiostats max out at $\sim \pm 10$ V and a few amperes; for anything beyond, specialized high-

voltage isolators or transformers are needed. Do not attempt to impedance-test a high-voltage module by directly connecting a low-voltage potentiostat – that's dangerous for the equipment and operator.

Thermal runaway risk: Although EIS signals are small, a fault or misuse could potentially heat a cell (for instance, if a short occurs or if one accidentally leaves the cell under a small DC bias). Always treat cells under test as if they could fail. For lithium batteries, have them in a tray or area that can contain any vent or leak. If testing in a temperature chamber, ensure the chamber has over-temperature protection. Never exceed the temperature limits of the cell (if a cell is rated 60 °C max, do not test at 80 °C unless it's part of a deliberate abuse test with proper precautions).

Pressure safety: If using high pressure in fixtures, be cautious of the stored energy. Use eye protection when assembling/disassembling high-pressure cells. Release pressure slowly to avoid parts springing out. Ensure bolts are tightened evenly to avoid uneven stress (which can cause a cell to crack or a bolt to shear). Use appropriate tools – e.g., a torque wrench – to not exceed fixture specs. Many fixtures have maximum force ratings; do not exceed them. If you need extremely high pressures (some solid-state research uses >100 MPa), that should be done in specialized presses with guarding.

Chemical exposure: If the interface zone involves hazardous materials (like sulfide solid electrolytes that release H₂S if exposed to moisture, or if a liquid electrolyte cell could leak), handle and dispose accordingly. Perform those tests in a fume hood or glovebox as required. Solid-state cells can contain ceramics (fine particles if broken) – avoid inhalation or skin contact.

Instrument compliance: Follow manufacturer's guidelines for calibration and maintenance of the EIS instrument. For compliance, instruments often adhere to standards like IEC 61010 (safety requirements for electrical equipment for measurement). While not directly your task to enforce, ensure your instrument has up-to-date safety certifications if needed (especially in a production setting, equipment should be certified).

Standards and testing protocols: Be aware of relevant testing standards. For example: - **IEC 62660-1:2010** (for automotive Li-ion cells) includes AC impedance at 1 kHz as part of performance tests 4 . If you are working with automotive cells, referencing this standard and possibly performing the 1 kHz test for comparison can be useful. That standard expects, say, internal impedance below a certain threshold for a cell to be acceptable 57 (e.g., one source noted <0.8 m Ω for some EV cells at 1 kHz 57). There are also **UL and UN** transportation safety tests (like UN38.3) but those involve things like vibration, not EIS. However, some general battery test standards (IEC 61960 for portable, etc.) mention storing data and doing reference performance tests that could include impedance.

If your lab or factory claims compliance with ISO 9001 or IATF 16949 (quality management systems), traceability and calibration will be part of those. Ensure all EIS measurements that inform decisions are done on calibrated equipment (keep records of calibration).

Environmental health and safety (EHS): In an EHS context, the focus is on safe operation of the testing and safe handling of results (like if data indicates a problem). For example, if EIS on a production cell shows unusually high impedance, it could indicate an internal fault (like a dry cell or developing internal corrosion). That cell might then be flagged as potentially hazardous (some failure modes correlate with increased impedance). Be prepared to remove such cells from normal processing and investigate safely.

Disclaimer on instructions: As the final note, this document is meant to provide general best practices and is based on published standards, application notes, and literature. It does **not** supersede your

internal protocols. Always contextualize these recommendations within your specific materials and setup. When in doubt, consult a specialist or the equipment manufacturer.

Now, before concluding, we provide in the Appendices additional resources: tables summarizing parameters and QA steps, a glossary of terms used, and an example data sheet template that could be adopted for recording EIS tests.

7. Limitations & Disclaimer

Limitations: The guidance provided is stack-agnostic and may need adaptation for specific battery chemistries or formats. While we strived to include values and procedures from authoritative sources (standards, peer-reviewed studies, established handbooks), some recommendations (such as specific waiting times or acceptance thresholds) are based on typical scenarios and may not cover edge cases. Different solid-electrolyte families or cell designs might present unique challenges (for instance, impedance of lithium metal interfaces can be highly nonlinear even at small amplitudes). Always consider additional literature specific to your system if available. In cases where sources conflict (e.g., one source might suggest a 10 mV amplitude, another 5 mV), we have reported ranges and reasoning so you can choose appropriately for your context 2 1.

Disclaimer: This report is intended for educational and planning purposes in a battery R&D and manufacturing context. It does not constitute an official standard or regulation. **Site-level standard operating procedures (SOPs) and safety protocols must take precedence** over any generalized advice given here. Implementation of any test method described should be done by trained personnel, and the authors and sources cited assume no liability for incidents arising from misuse. Always wear appropriate personal protective equipment (PPE) and ensure testing setups have fail-safes (fuses, etc.) where applicable. If this document is used as part of internal documentation, it should be reviewed and approved by the relevant engineering and EHS departments.

References

- Keysight Technologies Impedance Measurement Handbook, 6th Edition, Keysight
 Application Note, 2016. Guidelines on impedance measurement techniques, including open/short compensation and guarding.
 36 11 (Accessed 2025-09-19)
- 2. **Gamry Instruments Total Harmonic Distortion: Theory and Practice**, Gamry Application Note, 2019. Discussion of linear vs. non-linear response in EIS and the importance of AC amplitude (5–10 mV) to avoid distortion. (2) 15 (Accessed 2025-09-19)
- 3. Neware Technology Electrochemical Impedance Spectroscopy (EIS) News Article, Neware, 2024-06-26. Overview of EIS basics; notes linearity condition typically requires 5 mV amplitude (≤10 mV) and stability condition for valid EIS. 1 58 (Accessed 2025-09-19)
- 4. **Gamry Instruments EIS Measurement of a Very Low Impedance Lithium Ion Battery**, Gamry Application Note, 2010. Explains mutual inductance issues in 4-terminal EIS leads and how to minimize lead inductance (twisting, separating sense and current leads). 5 6 (Accessed 2025-09-19)
- 5. **Bio-Logic Science Precautions for good impedance measurements (EIS) Application Note** #5, Bio-Logic, 2023. Highlights error sources external to the cell (cabling, noise) and recommends shielding (Faraday cage) and guarding to reduce 50/60 Hz pickup and stray capacitance. 32 8 (Accessed 2025-09-19)
- 6. **Bio-Logic Science Two questions about Kramers-Kronig transformations Application Note #15**, Bio-Logic, 2009. Demonstrates K-K validation on impedance data and how truncated

- frequency ranges can be checked by fitting to a KK-consistent model (Voigt circuit). (Accessed 2025-09-19)
- 7. O. R. Bucci et al., "A guide to equivalent circuit fitting for impedance analysis and interpretation," Electrochimica Acta, vol. 182, pp. 89–102, 2015. Discusses pitfalls in equivalent circuit fitting; emphasizes no unique circuit for a given spectrum and caution against overinterpretation. 52 (Accessed 2025-09-19)
- 8. **Gamry Instruments Basics of Electrochemical Impedance Spectroscopy**, Gamry, 2010. Introductory tutorial on EIS; notes pseudo-linear behavior with 1–10 mV signals and warns that drift during long measurements leads to inaccurate results. ⁵⁹ ¹⁷ (Accessed 2025-09-19)
- 9. **Gamry Instruments Basics of EIS Part 2,** Gamry, 2010. Continuation covering equivalent circuits; advises using the fewest necessary elements and warns that adding elements can fit noise without physical relevance (empirical models with caution). ³ (Accessed 2025-09-19)
- 10. **IEST (Xiamen) Application of EIS Test to Lithium Battery Under Pressure,** IEST Case Study, 2025. Investigation of how external module pressure (0.27–2.7 MPa) affects pouch cell EIS; details frequency range 10 kHz–0.02 Hz, 5 mV amplitude, and 20 min stabilization at each pressure.

 44

 18 (Accessed 2025-09-19)
- 11. **Hioki Tier-1 Manufacturers Explore a Paradigm Shift in EIS Measurement,** Hioki Newsroom, Jun. 28, 2024. Industry perspective on using multi-frequency EIS for battery inspection; notes standard production test is 1 kHz AC IR, and benefits of extending to 0.01 Hz (near-DC) and intermediate frequencies (e.g. 100 Hz) for better insight. 60 47 (Accessed 2025-09-19)
- 12. MDPI Batteries Accelerated Internal Resistance Measurements of Lithium-Ion Cells..., J. Lamb et al., Batteries, 4(4), 49, 2018. Describes internal resistance measurement methods; references IEC standards (e.g. IEC 62660-1:2010) and discusses temperature, SOC, settling time impacts on internal resistance and EIS accuracy. 61 62 (Accessed 2025-09-19)
- 13. **IEEE IECON 2013 Validation of Impedance Data,** T. Schiller et al., IEEE Proceedings, 2013. Theoretical foundation for KK transforms; reiterates that comparing measured vs. KK-transformed real part tests self-consistency of EIS data. ²⁰ (Accessed 2025-09-19)
- 14. **Bio-Logic How to choose the proper equivalent circuit?**, Bio-Logic Learning Center, 2020. Guidance on model selection for EIS fitting, with emphasis on not overfitting and understanding physical meaning of elements. (Accessed 2025-09-19)
- 15. International Electrotechnical Commission (IEC) 62660-1, Secondary Lithium-Ion Cells for the Propulsion of Electric Road Vehicles Part 1: Performance Testing, IEC, 2010. Defines standard test methods for EV cells, including 1 kHz AC impedance measurement as internal resistance benchmark. (Accessed 2025-09-19)

(Note: Access dates refer to the date this report was compiled. Some sources have been paraphrased for clarity. In cases of web resources, the content cited was verified as of access date.)

Appendices

Appendix A: Example EIS Parameter Table

Table 1. Recommended Starting Parameters for EIS Measurement and Rationale

Parameter	Starting Value(s)	Rationale	Common Pitfalls	Source
AC amplitude (potentiostatic)	5 mV rms (≈10 mV p-p); not >10 mV	Ensures pseudo-linear response (minimizes harmonics) 1 . Large enough for good SNR in most cells.	Too low: noisy spectrum (esp. low freq); Too high: non-linear response (harmonics) 15 . Check linearity via Lissajous.	[3][2]
AC amplitude (galvanostatic)	Set current to yield ~5–10 mV across cell (e.g. $I = 0.1 \text{ A for } \sim 5 \text{ m}\Omega \text{ cell})$	Same reasoning as above, but for low-impedance systems a current excitation may be used. Keeps voltage perturbation small.	Too high current can drive cell into iR drop causing DC bias; too low current -> poor SNR. Ensure current < instrument limit.	[2]
Frequency range	100 kHz down to 0.1 Hz (decade steps)	Captures high- frequency resistances and low-frequency diffusion. 100 kHz is high enough for ohmic & inductive behavior; 0.1 Hz often shows diffusion tail.	Not low enough: miss slow processes; Not high enough: miss inductance or quick interface process. Very low (<0.01 Hz): long test, drift ¹⁷ .	[8][4]
Points per decade	~6 (log spaced)	Provides adequate resolution of impedance features without overly long test.	Too few: poor definition of arcs; Too many: unnecessary time, more data to noise-average (diminishing returns).	_
Integration time / point	≥1 cycle settle + 3–5 cycles measure (auto)	Averages out noise and ensures steady-state at each frequency. Longer at low freq to account for cell reaching periodic steady-state.	If too short, phase errors and scatter. If too long, test time lengthens and drift risk increases. Balance via trial runs.	_
Temperature (ambient example)	25 °C ±1 °C (controlled)	Standard reference temperature; small tolerance ensures consistency 61. Many impedance parameters vary ~2× per 10 °C (Arrhenius).	Uncontrolled lab temp can drift/fluctuate, causing run-to-run variance. If unable to control, at least record temp for each test.	[12]

Parameter	Starting Value(s)	Rationale	Common Pitfalls	Source
Temperature (elevated test)	e.g. 60 °C for life testing (±1 °C)	Higher temperatures sometimes used to accelerate processes – ensure chamber stability.	Condensation in humid env; thermal gradients in cell; instrument max temperature limits (some potentiostats not rated above certain temp unless remote sensing).	-
Pressure on cell (if applicable)	e.g. 5 MPa for solid-state stack (constant)	Many solid interfaces require pressure – choose relevant value (from literature or module design). Keep constant to compare results.	If not monitored, pressure might relax during test. Overtightening can damage cell. Use calibrated springs or torque with known conversion.	[10]
OCV rest time before EIS	≥2 hours (for full cell after cycling), ≥20 min (after pressure/ temperature set)	Allows electrochemical and thermal equilibrium 18. Ensures true OCV (no net reactions ongoing).	Insufficient rest leads to drift during test (violates steady-state) 17 . For high-capacity cells or after large SOC change, even longer rest may be needed (up to 24h).	[4][8]
Open circuit compensation	Perform before test, with fixture in place (no cell)	Measures stray capacitance admittance 11 – needed for accurate high-frequency data. Leaves instrument expecting 0 at open.	If skip, high-freq data includes unknown stray C (distorted slope). If done with different setup (distance), it over/ under compensates.	[1]
Short circuit compensation	Perform before test, shorting fixture electrodes	Measures residual series impedance 12 (lead resistance/ inductance) – subtracts it out from sample data.	Using a poor short (e.g. a wire with loops) adds inductance, leading to under-compensation. Ensure a direct, lowinductance shorting method.	[1]
Initial check frequency	1 kHz (or mid- band freq) at start/end	Quick single-frequency measurement to verify cell connection and stability. At 1 kHz many cells show mostly resistive behavior (for a rough internal resistance check) 4.	If large deviation between start vs end measurement, indicates drift or change during test – data quality in question. Use as QA flag.	[11]

Parameter	Starting Value(s)	Rationale	Common Pitfalls	Source
Reference electrode (3- elec) usage	N/A (if 2- electrode) or stable reference (e.g. Li metal in half- cell)	A reference can isolate an interface (for research). If used, ensure it's truly non- polarizable and placement yields low impedance reference impedance.	Poor reference (high impedance or drifting potential) will invalidate data (phase errors, etc.) 34. For solid-state, reference integration is complex; often 2-electrode is used for simplicity.	[5]

Sources: [1] Keysight Impedance Handbook 36 11 ; [2] Gamry THD note 15 ; [3] Neware EIS basics 14 ; [4] Gamry Low-Impedance app note 28 ; [5] Bio-Logic Precautions note 34 ; [8] Gamry EIS basics 59 63 ; [10] IEST pressure EIS 18 ; [11] Hioki industry report 4 ; [12] MDPI/Lamb et al. 61 .

Appendix B: QA Checklist Table

Table 2. QA Steps for Impedance Data and Acceptance Criteria

QA Step	Method	Pass / Tolerance Criteria	Artifact / Documentation	Source
Kramers– Kronig consistency check	Apply K-K transform test (software tool or modeling) on full dataset. Compare reconstructed vs. measured Z.	Pass: KK error < 5% (e.g. χ^2 or MAPE) across spectrum. No systematic deviation in Re/Im. **lnvestigate: 5–10% error or frequency-dependent mismatches. **strail: >10% error or non-physical trends (e.g. negative resistance seen).	Save KK analysis output (graph of measured vs. KK-fit). Note consistency metric in report. Example: "KK test: 2.3% error, passed."	[6][13]

QA Step	Method	Pass / Tolerance Criteria	Artifact / Documentation	Source
Repeat measurement (same sample)	Re-run EIS on same cell under identical conditions (immediately or after short interval).	Pass: Key parameters (R_s, R_ct, etc.) within ±5% or within instrument noise level. Overall Nyquist profile overlaps (no new features). <pre></pre>	Overlay impedance plots from both runs in a figure. Compute % difference of R values. Document: "R_ct was 45 Ω vs 48 Ω on repeat (6% diff) – acceptable."	_
Reproducibility (multiple samples)	Measure multiple nominally identical cells. Compare spectra or fitted parameters across batch.	Pass: All samples fall in expected range (e.g. R_ct spread ±10%). No outlier deviating beyond spec limit. 	Plot Nyquist of all samples or bar chart of key parameter for each. Statistical summary (mean, SD). Log any outlier serial numbers for follow-up.	_
High-frequency intercept sanity check	Check that the high-freq real axis intercept ~ equals known electrolyte/internal resistance (if independently measured by 1 kHz AC or pulse).	Pass: R_hf (from EIS \sim 10^5 Hz point) within \pm 10% of 1 kHz AC IR or DC ohmic drop measurement. 	Note in report: "High-frequency R = $5.2 \text{ m}\Omega$, vs 1 kHz AC IR = $5.0 \text{ m}\Omega$ (within 4%)." If fail, crosscheck instrument calibration or cell state.	[11]

QA Step	Method	Pass / Tolerance Criteria	Artifact / Documentation	Source
Residual inductance/ loop check	Examine highest-frequency data for inductive loop (Im(Z) positive). Also check if open/short comp eliminated >90° phase lead at high f.	Pass: No significant inductive loop beyond what's expected (e.g. <5° phase lead, smooth transition). Investigate: Small inductive hook present – likely minor lead inductance uncorrected, acceptable if noted. Fail: Large inductive artifact dominating highf (phase → +90°) – suggests bad wiring or no comp. Requires remeasure after fix.	Include comment in analysis: "High-f inductance ~10 nH observed, likely fixture – accounted in fit." If fail, attach photo of wiring and highlight needed change.	[4]
Low-frequency stability check	Compare the lowest freq point to the second-lowest (or repeat lowest freq twice). Also compare beginning vs end OCV.	Pass: Last two points align on trend (no jump), OCV shift < a few mV over test. Slight upward drift in final point – possibly minor ongoing process, acceptable if <5% effect. Fail: Significant upturn or OCV drift – indicates nonequilibrium, data at lowest freq not reliable.	Document OCV_before/after in lab notebook. Possibly exclude or annotate data below a cutoff frequency if drift detected ("Data below 0.2 Hz omitted due to instability").	[8]
Model fit quality (if circuit fitted)	Examine residuals of fit and physical sense of parameters. Possibly do chisquare of fit.	Pass: Residuals randomly scattered around 0 (no systematic shape). Fitted values within expected physical bounds (e.g. Cdl in μF/cm² range, n ~0.8-1). br>Investigate: Systematic residual (e.g. model can't capture midfrequency kink) – might need improved model or note limitation. br>Fail: Very large residuals or non-physical values (e.g. negative resistance) – model invalid for data.	Save fit report (parameters table and residual plot). Note in report if model is an equivalent circuit and give an Figure reference or diagram of it. Ensure no parameter is at a hard limit (if so, fit not trustworthy).	[9]

QA Step	Method	Pass / Tolerance Criteria	Artifact / Documentation	Source
Data logging completeness	Verify all metadata was recorded (per Section 5.1). Use a checklist or form.	Pass: Metadata form filled with sample ID, test conditions, etc. Any missing critical info (e.g. forgot to note temperature or which reference electrode was used) – try to recover from memory or logs, else mark data with caveat.	Conduct post-test review: an engineer or QA person cross-checks entries. E.g., ensure pressure value is listed if pressure applied. Store this form with data.	[15]

Sources: [4] Gamry low-impedance (lead inductance) 64 ; [6] Bio-Logic KK note 23 ; [8] Gamry EIS basics (steady-state requirement) 17 ; [9] Gamry model caution 3 ; [11] Hioki/IEC std (1 kHz IR) 65 ; [13] Schiller KK validation (passivity test) 20 ; [15] IEC 62660-1 (metadata context for traceability) 53 .

Appendix C: Glossary of Terms

- AC Amplitude (EIS): The magnitude of the oscillatory perturbation applied during EIS, either as a voltage (potentiostatic EIS) or current (galvanostatic EIS). Kept small to ensure linear system behavior 1. Typical values: 5–10 mV or such that the current response is in the linear regime 15.
- Causality (in EIS context): The principle that the output (current) at any time is only caused by prior or present inputs (voltage), not future ones. In EIS, this is assumed; violation can occur if data is not physically meaningful. K-K relations derive from causality 20.
- Constant Phase Element (CPE): A non-ideal capacitor element with impedance $Z=[Q(j\omega)^n]^{-1}$. If n=1, it behaves like a pure capacitor with Q = C (F). If n<1, it represents a distribution of time constants (often due to rough surfaces or heterogeneous interfaces). Used in equivalent circuits to model depressed semicircles.
- Electrode | Electrolyte Interface: The boundary region between an electrode (anode or cathode) and the electrolyte (liquid, polymer, or solid) where charge transfer reactions occur. In solid-state batteries, often refers to the contact between a solid electrode and solid electrolyte. Impedance of this interface typically includes charge-transfer resistance and double-layer capacitance (or CPE) in parallel.
- Four-terminal (4T) sensing: Same as Kelvin sensing; uses separate pairs of leads for current and voltage measurement to eliminate lead/contact resistance from measurements ²⁵. Essential for low-impedance measurements (battery cells, interface stacks) to get accurate EIS.
- **Guarding:** A technique to eliminate effects of stray capacitances by surrounding a sensitive node with a guard conductor held at nearly the same potential, thus preventing leakage currents from affecting measurements ⁹ ¹⁰ . Many EIS instruments have a guard terminal (connected to cable shields) to implement this.

- Kramers-Kronig (KK) Relations: Mathematical integrals linking the real and imaginary parts of a complex function that is linear, causal, and stable. In EIS, KK relations provide a consistency check

 any physically valid impedance spectrum must obey them ²⁰. Violations indicate issues like drift or measurement error.
- Linear System (pseudo-linear in EIS): A system is linear if its output is directly proportional to input (following superposition). Electrochemical systems are inherently non-linear (e.g. Butler-Volmer kinetics) but can be approximated as linear by using a sufficiently small perturbation around a steady state 2. "Pseudo-linear" implies linear behavior in response to the small AC, even if the overall V-I curve is non-linear.
- Nyquist Plot: A common way to display EIS data with the real part of impedance (Z') on the X-axis and negative imaginary part (–Z'') on the Y-axis. Each point corresponds to a frequency (high freq on left, low freq on right). Semicircular features often indicate parallel R–C processes; a 45° line indicates Warburg diffusion.
- OCV (Open-Circuit Voltage): The voltage of the cell when no current flows (equilibrium). In impedance testing, the cell is usually held at OCV (no DC bias) unless a specific bias is being studied. A stable OCV is needed to consider the system at steady-state for measurement 66.
- Open/Short Compensation: Calibration steps where an impedance analyzer measures an open circuit (infinite impedance) and a short circuit (nearly zero impedance) to characterize its own parasitic admittance and impedance. These are then subtracted from sample measurements to improve accuracy 36 12.
- Passivation Layer / SEI: A solid layer that forms at interfaces (e.g., Solid Electrolyte Interphase on anodes, or cathode electrolyte interphase on cathodes). These layers often introduce additional resistance and capacitance in the impedance spectrum. For instance, a high-frequency small semicircle is sometimes attributed to an SEI film resistance and capacitance.
- Pressure (Stack Pressure): The mechanical pressure applied to a cell or stack, particularly relevant in solid-state batteries to maintain interfacial contact. Measured in MPa (or equivalently N/m^2). Changes in pressure can alter interface impedance significantly 19.
- Repeatability vs. Reproducibility: Repeatability refers to variation when the same person/ equipment measures the same sample under same conditions repeatedly (short-term). Reproducibility refers to variation when conditions change, e.g. different operator or instrument, or over longer term or across nominally identical samples. In context, we want EIS to be repeatable (low intra-sample variation) and reproducible across similar samples.
- Residuals (fitting): The difference between measured impedance data and the model impedance (from an equivalent circuit) at each frequency. Plotted typically as $\Delta Z'$ and $\Delta Z''$ vs freq. Random residuals around zero indicate a good fit; structured residuals indicate model deficiency.
- Warburg Impedance: Impedance with a 45° phase angle, characteristic of diffusion-controlled processes. In finite-length diffusion, it transitions to a vertical line (capacitive behavior) at low frequencies, often modeled as a finite Warburg element or Gerischer. The presence and slope of a Warburg can tell diffusion coefficient info.

- 1 kHz AC Resistance (ACIR): A quick measurement of internal resistance using a 1 kHz AC signal. Used in many standards (like IEC 62660-1) and battery testers for quality control 65. It effectively measures the high-frequency intercept of impedance (since at 1 kHz, capacitive reactance of double-layer is usually low, so it approximates ohmic + some charge transfer). It's not the full spectrum, but often correlated with cold cranking ability in 12V batteries, etc.
- Battery Passport: An emerging concept (particularly in EU regulations) requiring detailed data recording for batteries through their life cycle 53. While not an EIS term, it underscores the importance of data traceability and standardization, meaning your EIS results might eventually feed into such a data profile for cells.

2 15 40 Total Harmonic Distortion: Theory and Practice Gamry Instruments https://www.gamry.com/application-notes/EIS/total-harmonic-distortion/
3 17 45 59 63 66 Basics of EIS: Electrochemical Research-Impedance Gamry Instruments https://www.gamry.com/application-notes/EIS/basics-of-electrochemical-impedance-spectroscopy/
4 16 24 46 47 53 54 60 65 Tier-1 Manufacturers Explore a Paradigm Shift in EIS Measurement for High-Capacity Batteries Hioki https://www.hioki.com/us-en/news/detail/id_n1266955
5 6 25 26 27 28 29 30 31 64 Battery Impedance Modeling-EIS Measurement Low Impedance LIB Gamry Instruments https://www.gamry.com/application-notes/EIS/eis-measurement-of-a-very-low-impedance-lithium-ion-battery/
7 8 32 34 35 Precautions for good impedance measurements (EIS) Battery & Electrochemistry - Application Note 5 - BioLogic https://www.biologic.net/documents/eis-precautions-electrochemistry-battery-application-note-5/
9 10 11 12 13 33 36 37 38 39 cmc.ca https://www.cmc.ca/wp-content/uploads/2019/07/Keysight-Technologies-impedance-measurement-handbook.pdf
18 19 44 EIS Testing Of Lithium Batteries Under Pressure Condition https://iestbattery.com/case/eis-test-for-lithium-batteries-under-pressure/
20 21 deutronic.de https://www.deutronic.de/wp-content/uploads/2021/02/Validation-of-Impedance-Data.pdf
22 23 49 50 51 EC-Lab - Application Note https://www.biologic.net/wp-content/uploads/2019/08/battery-eis-kramers-kronig_electrochemistry-an15.pdf
41 42 57 61 62 Accelerated Internal Resistance Measurements of Lithium-Ion Cells to Support Future End-of-Life Strategies for Electric Vehicles https://www.mdpi.com/2313-0105/4/4/49
Research on electrochemical impedance spectroscope behavior of https://www.sciencedirect.com/science/article/abs/pii/S0360319921007126
48 Electrochemical Impedance Spectroscopy A Tutorial https://pubs.acs.org/doi/10.1021/acsmeasuresciau.2c00070

55 Hierarchical Representation of Measurement Data, Metrological ...

https://chemistry-europe.onlinelibrary.wiley.com/doi/10.1002/batt.202300514

52 Typical equivalent circuit elements for EIS spectra analysis

⁵⁶ Accurate EIS Testing with Keysight Battery Test Systems

1 14 58 Electrochemical Impedance Spectroscopy(EIS)

https://www.neware.net/news/electrochemical-impedance-spectroscopy/230/70.html

https://www.keysight.com/blogs/en/tech/bench/2025/04/28/accurate-eis-testing-with-keysight-battery-test-systems

 $https://www.researchgate.net/figure/Typical-equivalent-circuit-elements-for-EIS-spectra-analysis_tbl1_288008115$