

# > In-Situ SEM Bulk Liquid Electrochemistry

## Technical Specs



	1470 Series SEM
<b>Total Electrodes</b>	6
<b>True Reference Electrode</b>	Yes*
<b>True Counter Electrode</b>	Yes*
<b>Electrolytes</b>	Aqueous, Wide range of organics
<b>Spacer Range</b>	100 nm to 2 um*
<b>Heating Compatibility</b>	Yes
<b>SEM Compatibility</b>	Custom integration

*\* Different Configurations and Materials Available*

## Features

### Overview

Hummingbird Scientific's dedicated SEM bulk liquid electrochemistry specimen holder, similar to the transmission electron microscopy (TEM) and X-ray Microscopy (XRM) counterparts, uses a real reference electrode (e.g., Ag/AgCl in KCl) and a real counter electrode (e.g., Pt) which allow the user to perform realistic in-situ electrochemistry that is for the first time quantitatively accurate and directly correlates to the industry-scale cells' electrochemical behavior. This is vastly different from the conventional in-situ liquid cells that use pseudo reference electrodes which are unreliable and present inaccurate electrochemical data. Key characteristics of a new in-situ specimen holder include:

- Replication of electrochemical data to industry-scale cell
- Observation of transient electrochemical behavior in realtime.

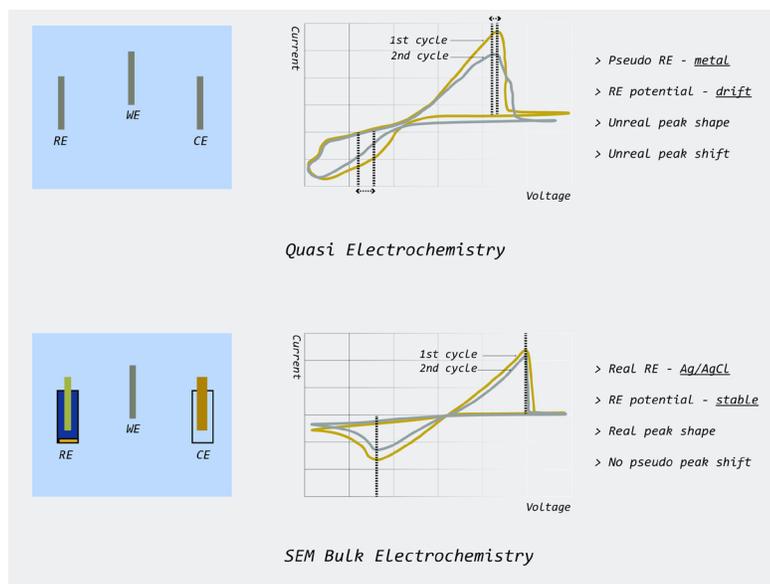
- Applications:**
- > Batteries
  - > Fuel cells
  - > Electrosynthesis
  - > Electrolysis
  - > Corrosion



## How it Works

The scanning electron microscopy (SEM) bulk liquid electrochemistry specimen holder is the first and only in-situ liquid cell solution for SEM with capabilities allowing true reference and counter electrodes performance for electroanalytical measurements. The electrodes' stability with little or no interference with the working electrodes allows superior performance and accuracy in the electroanalytical measurements.

The comparison of cyclic voltammetry – (current-voltage) data between a quasi electrochemistry system and our newest SEM bulk electrochemistry specimen holder is shown in Figure (left). The quasi electrochemistry platform with unreal reference (e.g., metal) and unreal counter electrodes result in redox curves that do not have characteristic shapes, and peaks show artificial shifts during cycling. The new SEM bulk electrochemistry holder with real reference electrodes (e.g., Ag/AgCl (in KCl) and real counter electrodes (e.g., Pt) show distinct peaks and no pseudo peak shift, replicating data (for the first time), relevant only in true bulk-scale electrochemical systems.



## Cross-Correlative



Along with the SEM system, our bulk liquid electrochemistry specimen holder is also available for transmission electron microscopy (TEM), and X-ray microscopy (XRM). The in-situ characterization made possible from the multi-modal platform provides complementary datasets suitable for accurate and reliable electroanalytical measurements of the sample at various length scales, and with complementary imaging and spectroscopy techniques.

## Related Products



### ▶ Bulk Liquid Electrochemistry TEM Holder

Bulk liquid electrochemistry TEM holder uses real reference and counter electrodes to accurately perform quantitative electroanalytical measurements in TEM in-situ in any liquid medium.



### ▶ X-Ray/Synchrotron Liquid Holder

Hummingbird's synchrotron holder is a complete in-situ x-ray lab system, enabling high-resolution material characterization in liquids. The system offers single-inlet, heating, electrochemical, spectroscopy, and cross-correlative features.



### ▶ Gas Flow TEM Holder

Using the gas flow holder, researchers can characterize material behavior in gas environments at elevated temperatures and observe gas-solid interactions at high resolution. The holder is available with either a single or multi-channel gas delivery system.

## Featured Research

### Quantitatively tracking electrodeposition of catalyst particles in SEM in real-time

Researchers at the Fritz-Haber Institute of the Max Planck Society used Hummingbird's bulk liquid electrochemistry specimen holder in the SEM to demonstrate an accurate and consistent way to deposit copper oxide catalysts in various shapes and sizes. The study made use of the reliable reference electrode system incorporated in the bulk liquid system to quantitatively track the electrodeposited copper cubes with different facets and sizes over many (repeatable) cycles. For example, the growth starts from nucleation during the first cycle and deposition and dissolution of selective crystals occurs in the third and subsequent cycles. The work provides critical insights into developing catalysts to convert carbon dioxide into useful chemicals and fuels.

**Figure:** Current-voltage curve tracking oxidation and reduction peaks of copper oxide in different concentrations of KCl solution in 5 mM copper sulfate. Growth of Cu cubes and generation/dissolution of noncubic particles.

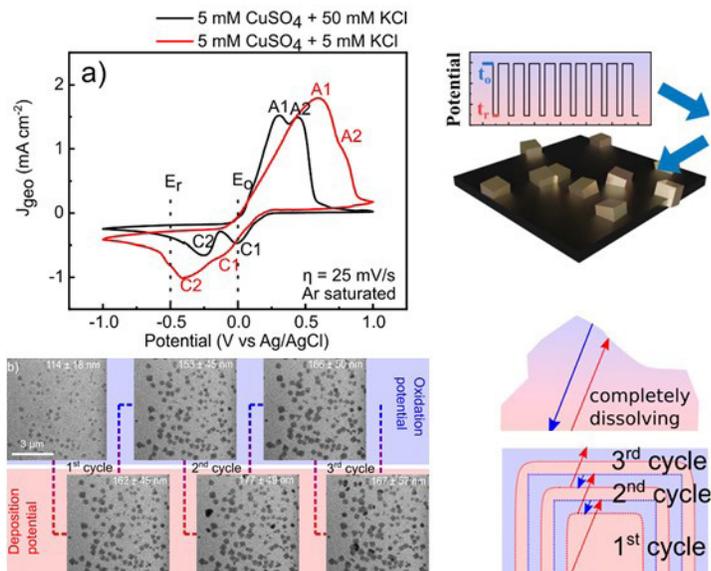


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**Reference:** Grosse et al. *J. Phys. Chem. C* 2020, 124, 49, 26908–26915.

DOI: 10.1021/acs.jpcc.0c09105

## Accessories



Accessories available for your SEM Liquid Holder include:

- Bulk Reference Electrodes – Any Research Standard or User's Choice
- Bulk Counter Electrodes – Any Research Standard or User's Choice
- Specialized Liquid Electrochemistry Chips
- Custom SEM Seal-Checking Station
- Liquid-Heating Controller
- Vacuum Tip Cover

## Selected Publications

Aram Yoon, Antonia Herzog, Philipp Grosse, Daan Hein Alsem, See Wee Chee and Beatriz Roldán Cuenya. **Dynamic Imaging of Nanostructures in an Electrolyte with a Scanning Electron Microscope.** *Microscopy and Microanalysis* (2021)

Philipp Grosse, Aram Yoon, Clara Rettenmaier, See Wee Chee, and Beatriz Roldan Cuenya. **Growth Dynamics and Processes Governing the Stability of Electrodeposited Size-Controlled Cubic Cu Catalysts.** *The Journal of Physical Chemistry C* (2020)

For the most up-to-date Selected Publications please visit: <https://hummingbirdscientific.com/products/sem-bulk-liquid-echem/>



Visit our Website



2610 Willamette Dr NE  
Lacey WA 98516  
t: 360.252.2737 - f: 360.252.6474

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