

> Bulk Liquid Electrochemistry

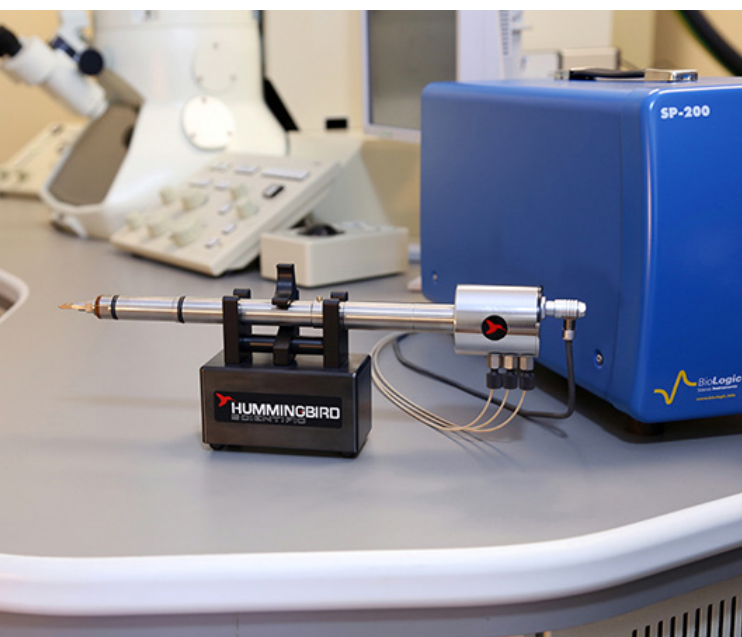
Technical Specs



	1490 Series
Total Electrodes	6
True Reference Electrode	Yes - drift rate less than 0.1 mV/min
True Counter Electrode	Yes
Counter Electrode Material	User's choice of material
Electrolytes	Aqueous, Wide range of organics*
Spacer Range	100nm to 2 μm^*
Heating Capability	Yes
EELS/EDS Compatible	Yes
TEM Compatibility	TFS, JEOL, Hitachi

* Contact us for Custom Configurations

Overview



Hummingbird Scientific was the first to sell a commercial liquid-electrochemical TEM holder in 2008. With the release of the Generation V in-situ bulk electrochemical TEM specimen holder, we are now providing the first and only in-situ holder that can accurately replicate bulk electrochemistry behavior of a wide range of liquid-electrochemical systems in-situ. The holder can measure bulk level electrochemistry data in-situ using up to six electrodes, a wide range of bulk materials on the CE, and a variety of real reference electrode options for various electroanalytical modes of operation.

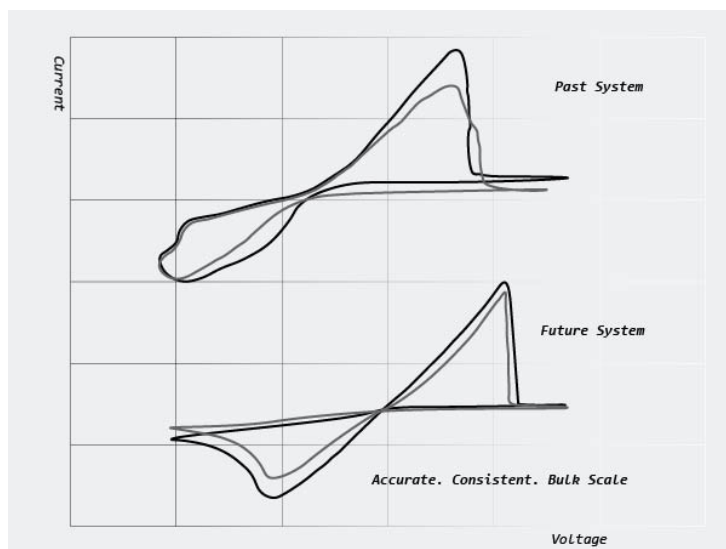
Sample Applications:

- > Electrochemistry
- > Corrosion
- > Electroplating
- > Electrolysis
- > Battery Materials
- > Fuel Cell Materials

How it Works

The new Generation V liquid-electrochemistry holder is the first ever liquid solution for TEM with capabilities allowing true reference and counter electrodes performance for electro-analytical measurements. The stability of the electrodes with little or no interference with the working electrodes allows superior performance and accuracy in the readings during the electroanalytical measurements.

The comparison of cyclic voltammetry data between a typical standard liquid holder and newest generation of liquid-electrochemistry holder shows remarkable improvement in the latter with distinct and consistent redox peaks over two cycles in a plating experiment (Figure on the left). The quality of data in the Generation V liquid-electrochemistry holder matches the performance of the standard beaker electrochemistry.

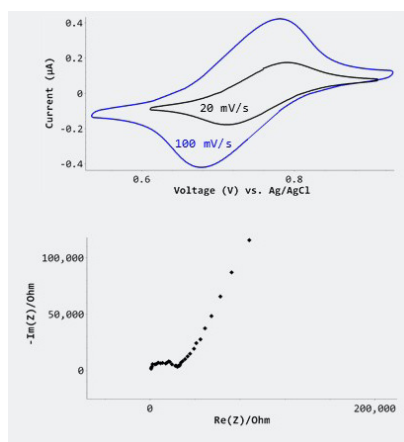


Model Baseline Electrochemistry – Ferrocyanide/Ferricyanide Redox

The Generation V liquid-electrochemistry in-situ TEM holder uses a completely newly developed hardware system and optimized electrochemistry chips with configuration of working electrode (WE), counter electrode (CE) and reference electrode (RE) that for the first time replicate bulk electrochemical conditions in-situ in the TEM. This was validated using several model electrochemical systems.

We performed cyclic voltammetry studies in a model 20 mM $K_3(Fe(CN)_6)/20$ mM $K_4Fe(CN)_6$ in 0.1M KCl solutions. The redox reaction of 20 mM ferrocyanide/ 20 mM ferricyanide in 0.1M KCl at different voltage scans show reversible electrode reaction during both the forward and reverse scans, elucidating bulk behavior. The corresponding electrochemical impedance spectroscopy (EIS) measurements shows lower capacitive current, and better signal-to-noise ratio with the lower concentration of solution.

HBS internal data obtained in collaboration with William C. Chueh group at Stanford University.

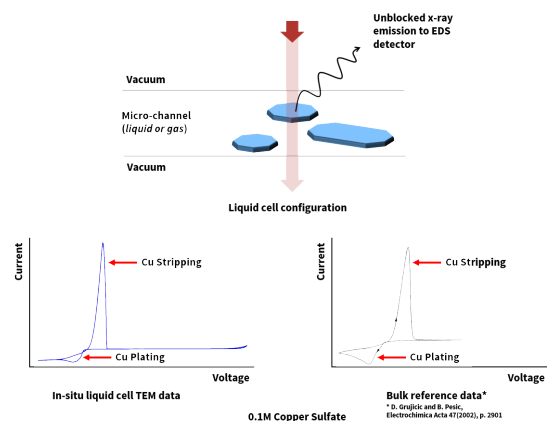


Top shows CV cycle performed at various potential scan rates – 20 mV/s and 100 mV/s. Bottom shows the corresponding EIS spectrum.

Model Baseline Electrochemistry – Cu Electrodeposition

To validate the performance of the Generation V Bulk Electrochemistry holder, we also perform TEM copper electrodeposition and compare the behavior with the beaker level bulk data.

A cyclic voltammetry (CV) study of a model compound 0.1 M $CuSO_4$ showing plating and stripping of copper has been presented here. The copper plating and stripping occur at the Pt working electrode at distinct redox peaks and the result replicate the bulk electrochemical cells with large electrode areas and larger volume of electrolyte solution.



Comparison of CV curves between in-situ TEM and bulk reference data.

Available For

ThermoFisher
SCIENTIFIC

THERMO FISHER SCIENTIFIC TEM

HITACHI HITACHI TEM

JEOL JEOL TEM

TEM Safety

Careful preparation of your samples and system are essential for effective use of environmental holders. A critical component of any holder system is a high-vacuum leak check station.

Our high-vacuum pumping station is a compact, all-in-one vacuum storage and seal-checking mechanism for TEM specimen holders. The station features short pumping and venting times, a low base pressure (1×10^{-6} mbar), and a glass viewing port for the holder tip.



Accessories



Accessories available for your Bulk Liquid Electrochemistry holder include:

- > Specialized Liquid Cell Chips
- > Leak Checking Station
- > Liquid-Heating Controller
- > Vacuum Tip Cover

Related Products



> **Liquid Holder**

The liquid holder is a complete in-situ TEM lab system, enabling high-resolution material characterization in liquids. The system offers single-inlet, dual-inlet, heating, electrochemical, spectroscopy, and cross-correlative features for your research.



> **X-Ray/ Synchrotron Liquid Holder**

The 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 for your research.



> **Electrochemistry Suite**

Hummingbird Scientific offer a comprehensive suite of holders for electrochemical battery research across various microscopy platform

Featured Research

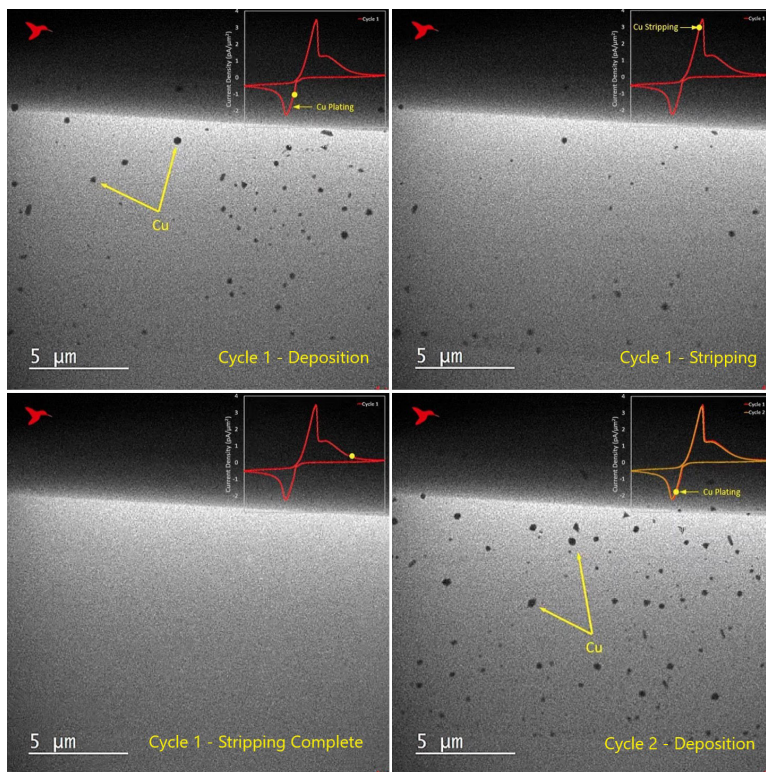
Replicating bulk electrochemistry experiments in-situ in the TEM

Hummingbird Scientific's newest Generation V Bulk in-situ liquid-electrochemistry holder allows researchers for the first time to fully replicate bulk-level electrochemical details inside the transmission electron microscope. A newly developed hardware system allows quantitative measurements of electrochemical processes with details showing the complete cycle of the bulk.

As an illustration, a cyclic voltammetry (CV) study of a model compound CuSO_4 solution showing plating and stripping of copper from the active electrode has been presented here. The copper plating and stripping occurs at the working electrode, replicating actual bulk behavior.

The new Hummingbird Scientific's liquid-cell hardware and cells allows, for the first time, for replication of bulk-level data in TEM.

HBS internal data obtained in collaboration with Rui Filipe Serra Maia and Eric Stach at the University of Pennsylvania.



In-situ liquid cell TEM copper plating and stripping from CuSO_4 solution. Insert chart's yellow dot shows corresponding CV curve position.

Selected Publications

Khim Karki, Rui Serra-Maia, Eric Stach, Daan Hein Alsem, Norman Salmon. **"Realistic Bulk Electrochemistry in Liquid Cell Microscopy."** *Microscopy & Microanalysis* (2020)

Nikhilendra Singh, Timothy S. Arthur, Oscar Tutusaus, Jing Li, Kim Kisslinger, Huolin L. Xin, Eric A. Stach, Xudong Fan, and Rana Mohtadi. **"Achieving High Cycling Rates via In-situ Generation of Active Nanocomposite Metal Anodes."** *ACS Applied Energy Materials* (2018)

Mei Sun, Xing Li, Zhiqiang Tang, Xianlong Wei and Qing Chen. **"Constant-Rate Dissolution of InAs Nanowires in Radiolytic Water Observed by In situ Liquid Cell TEM."** *Nanoscale* (2018)

Khim Karki, Tyler Mefford, Daan Hein Alsem, Norman Salmon, and William C. Chueh. **"Replicating Bulk Electrochemistry in Liquid Cell Microscopy,"** *Microscopy & Microanalysis* (2018)

Khim Karki, Daan Hein Alsem, and Norman Salmon. **"Practical Electroanalytical Measurements in Liquid Cell TEM,"** *Microscopy & Microanalysis* (2018)

Jeung Hun Park, Tommy Watanabe, Ainsley Pinkowitz, David J. Duquette, Robert Hull, Daniel A. Steingart and Frances M. Ross. **"In situ EC-TEM Studies of Metal Thin Film Corrosion in Liquid Solutions at Elevated Temperatures."** *Microscopy & Microanalysis* (2018)

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