

# Experiment Brief

## K3 IS Camera and 626 Liquid Nitrogen Cryo-Transfer Holder

### Title

Imaging discrete ions at a liquid-solid interface using low-dose cryo-electron microscopy (cryo-EM) and electron counting.

### Gatan Instrument Used

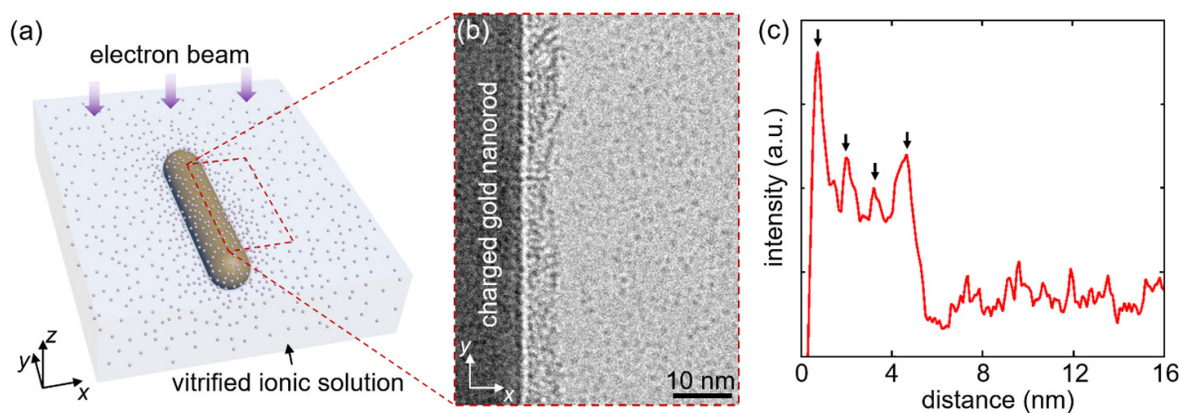
The K3<sup>®</sup> IS camera delivers simultaneous **low-dose imaging via real-time electron counting**, fast continuous data capture, and a large field of view. The Model 626 Liquid Nitrogen Cryo-Transfer Holder enables **frost-free low-temperature specimen transfer** and **subsequent imaging of radiation-sensitive frozen samples at high resolution** with precise temperature measurement.

### Background

Cryo-EM imaging has revolutionized structural biology by enabling imaging of proteins and other biomolecules at near-atomic resolution. Now, the same low temperatures and electron dose rates are being applied to other aqueous materials. In this case, the liquid-solid interface between a positively charged gold nanorod and an aqueous solution of phosphotungstic acid was studied to provide insight into the individual and collective behavior of ions at that interface. Interactions between a solid surface, ions, and the solvent govern the behaviors of a wide variety of technologically relevant materials, including batteries, catalysts, colloids, membranes, and supercapacitors, so understanding those interactions is essential.

### Materials and Methods

The sample was prepared by plunge-freezing a thin liquid film on a lacey carbon TEM grid. The temperature was maintained at 100 K as the sample was inserted into an image-corrected Titan ETEM using a model 626 cryo-holder from Gatan. Images were collected with a K3 IS camera in counted mode with a dose rate of  $500 \text{ e}^-/\text{\AA}^2/\text{s}$  and a total dose of  $100 \text{ e}^-/\text{\AA}^2$ .



**Figure 1.** Imaging ions at a liquid-solid interface. a) Experimental setup. b) Experimental image. c) Profile from the red region in b showing the presence of distinct layers. Arrows indicate the position of ion peaks predicted using a molecular dynamics simulation. Adapted with permission from Li, H.-K., et al. *Nano Lett.* 2020, 20 (11), 7927–7932. <https://doi.org/10.1021/acs.nanolett.0c02669>. Copyright 2020 American Chemical Society.

### Summary

The combination of low-dose imaging using the counted mode of the K3 camera and low temperature achieved using the model 626 holder enabled imaging of the ion distribution around a liquid-solid interface, with individual ions clearly resolved. Linear profiles and their FFTs were used to analyze the images, and this analysis found discrete layering of ions adjacent to the surface, as well as ordering along the layers. These were additionally compared with molecular dynamics and image simulations to aid in the experimental image interpretation.

### Credit(s)

A special thanks to Stanford University, including Hao-Kun Li, Ze Zhang, Joel Martis, and Arun Majumdar.

**Gatan, Inc.** is the world's leading manufacturer of instrumentation and software used to enhance and extend electron microscopes—from specimen preparation and manipulation to imaging and analysis.

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