

How to get clear images in Scanning Vibrating Electrode Technique (SVET)*

Version 1.0

*Advice made throughout this tutorial can be applied to SVET measurements on the M470, and M370 instruments



This tutorial aims to provide users with the information they need to obtain clear images using Scanning Vibrating Electrode Technique (SVET, also known as Vibrating Probe and Scanning Vibrating Probe; SVP). We will address a number of factors which can affect the SVET image:

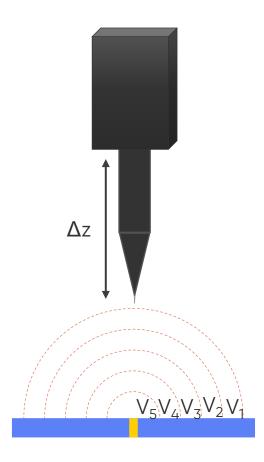
- The sample
- The electrolyte
- Sample tilt and topography
- Configuration settings

Once mastered users will be able to measure both model and novel samples.

The Experiment Components.

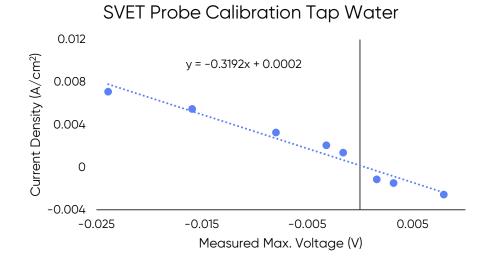
A note on the probe.

Variations in the tip can affect the collection efficiency of each probe. As a result each probe must be individually calibrated under experimental conditions to correlate the measured signal with current density.



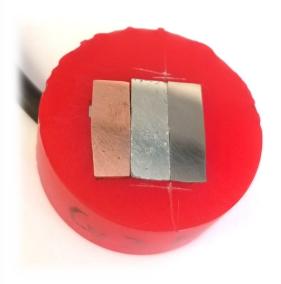
Probe calibration.

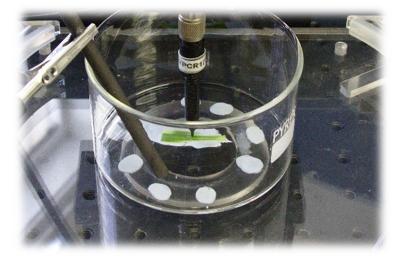
- In SVET the raw data is voltage
- The probe is calibrated to determine the current density
 - Unless otherwise stated the calibrated data is shown in this tutorial
- To calibrate the probe the current density of a Point In Space (PIS) is measured at different bias voltages
- The calibration process is detailed in <u>SCAN-Lab TN#2</u>



Sample requirements.

- Must be naturally active (e.g. corroding, living) or biased
- An electrical connection is beneficial but not always required



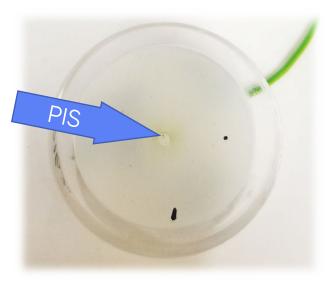




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A note on the sample.

- In this tutorial experiments are performed on a 200 μm Au Point In Space (PIS) sample, unless otherwise stated
- The PIS was biased using an SP-300 controlled by the M470 software



Mounting the sample.



Blu-Tac

Can be used to hold small (few cm) samples onto a blank.

Sample s

Sample should not be porous, or adversely affected by heating.



Epoxy Resin

Should only be used for samples which are polished before measurement.



Other

PTFE tape, parafilm, double sided tape, glue, adhesive medical tapes... In SVET an electrical contact to the sample is advised, even for naturally active samples, as this improves the signal strength measured.



Soldered wire

Particularly useful for epoxy mounted samples which will be used repeatedly.



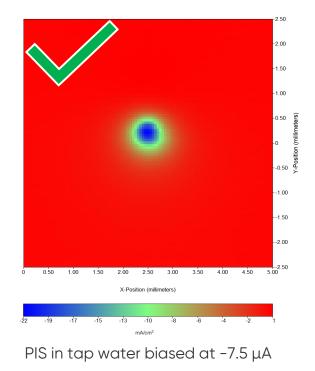
Copper tape

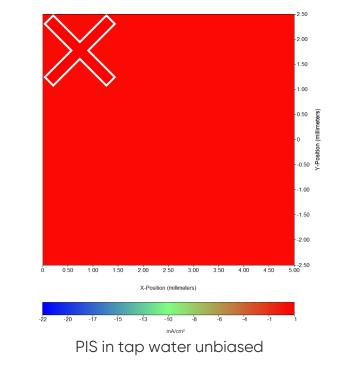
Useful for samples not easily soldered to, or connected to with a crocodile clip. Often requires use of silver paint.



Crocodile clip

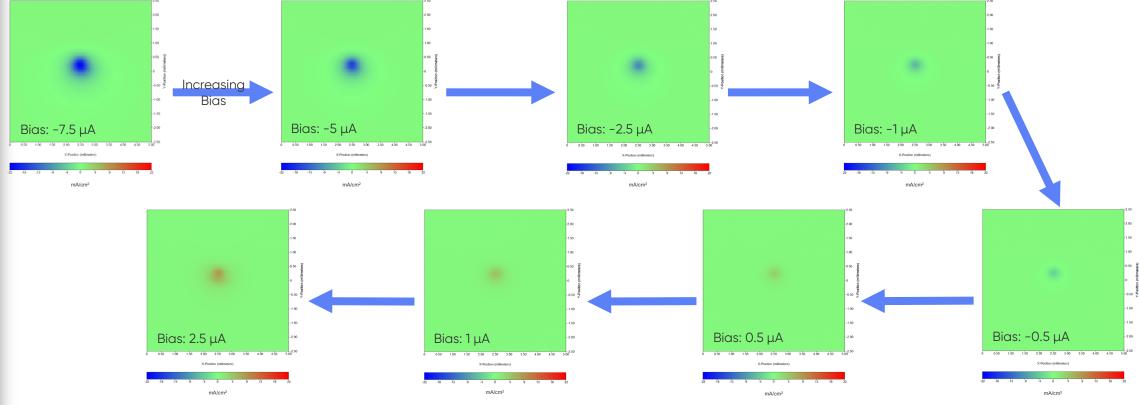
For large flat samples it may be possible to directly connect to the sample using a crocodile clip. In SVET if the sample is not naturally active, or has low natural activity it should be biased, otherwise the signal will be weak/non-existent.







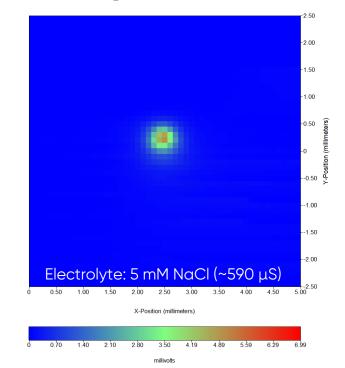
As the bias current applied to the sample changes the measured current density changes, with stronger signals associated with larger magnitude bias currents

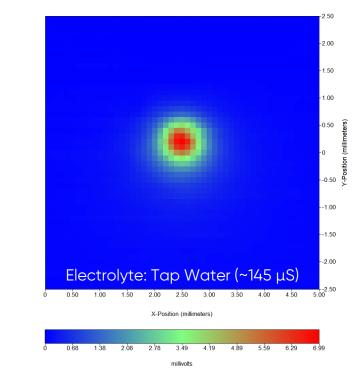


PIS measured in tap water with changing bias

The electrolyte.

- SVET signal strength depends on electrolyte conductivity
- The lower the conductivity the stronger the voltage response at the same probe to sample distance

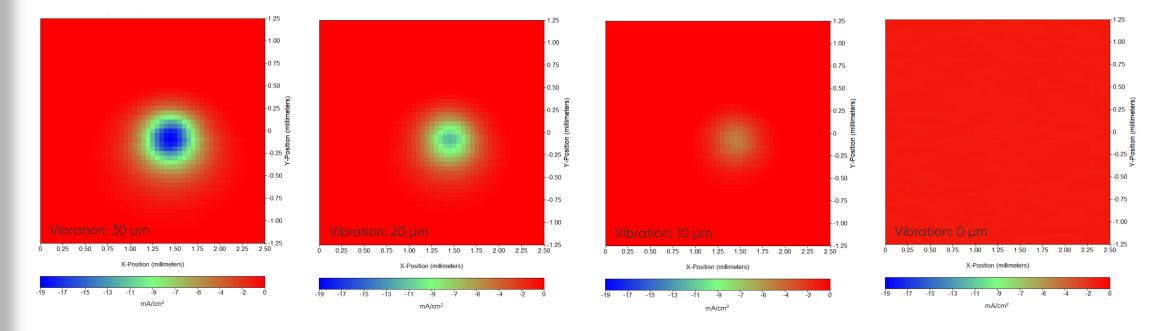




Note: For clarity the raw SVET data is shown

Probe vibration.

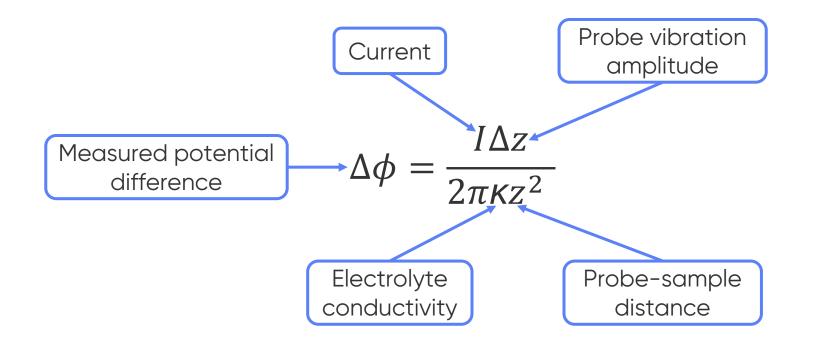
As the probe vibration decreases the signal strength also decreases



PIS measured in tap water with -5μ A bias

Key factors affecting SVET signal.

The measured SVET signal is defined by the equation below:

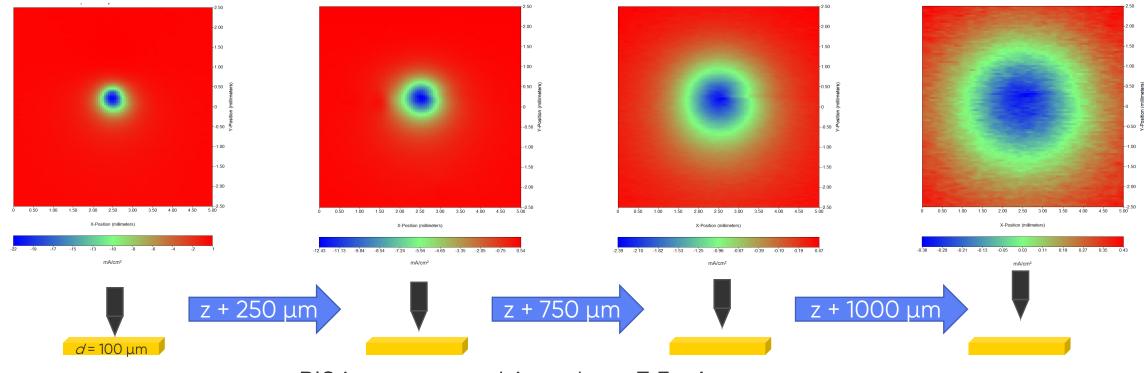


For more information see: <u>SCAN-Lab TN#8: Scanning Vibrating Electrode Technique (SVET): factors</u> affecting the measurement

Sample tilt and topography.



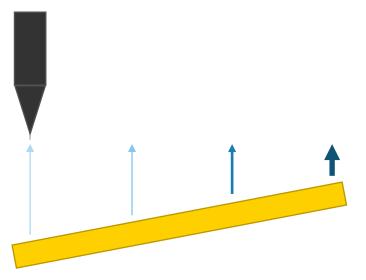
- To measure a high quality signal the probe should be near the surface (~100 $\mu m)$
- It should not touch the surface at any point in the scan



PIS in tap water biased at $-7.5 \,\mu\text{A}$



- Too much sample tilt causes partial or complete loss of SVET signal.
- Too much sample tilt can reduce the signal quality and distort the image
- Sample tilt can lead to probe crash.



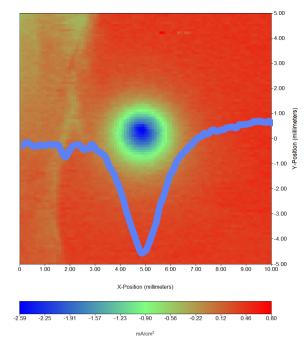
Dealing with sample tilt: During setup.

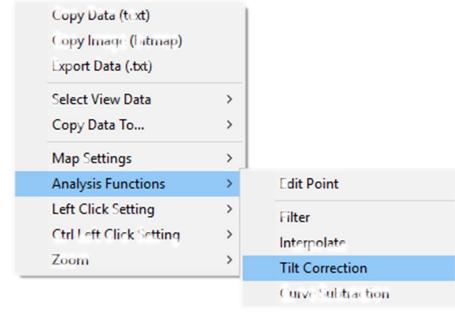
A spirit level is supplied to help level the sample in the TriCell using the adjustment screws.

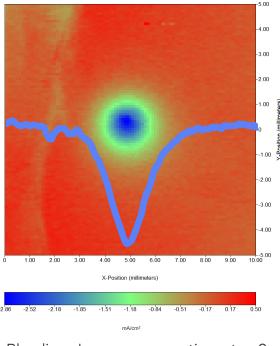


Dealing with sample tilt: Post experiment.

If sample tilt is apparent in an area scan it may be possible to correct for this in the M470 and M370 software.







Blue line shows cross section at y=0

Blue line shows cross section at y=0

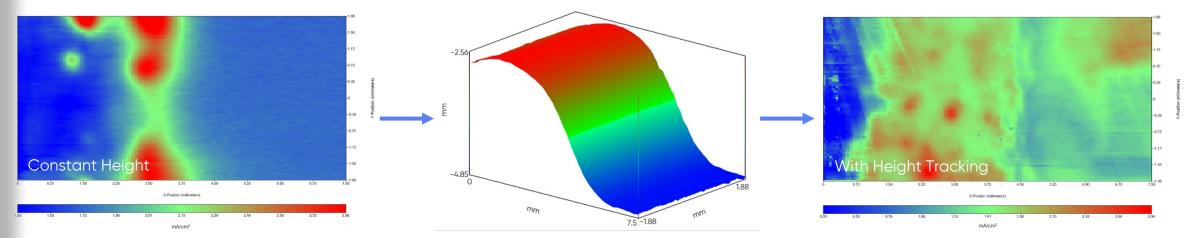
PIS in tap water biased at $-7.5 \,\mu\text{A}$

Dealing with sample topography.

The SVET signal depends on sample topography. To reduce topography effects the probe can track the sample using Height Tracking (HT) with topography measured by the <u>Optical</u> <u>Surface Profiler (OSP)</u> technique first.



Steel weld measured at Open Circuit Potential (OCP) in 5 mM NaCl with and without height tracking



Topography from OSP

Tips for height tracking.

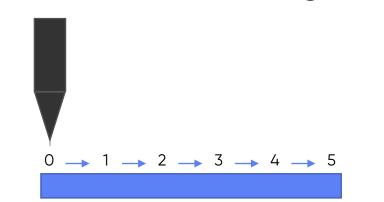
- Confirm the measured topography matches expectations
- Bad topography data causes bad SVET data
 - Spurious points should be edited
 - If the topography data is very poor re-run the experiment

Configuration settings.

In constant height SVET both step scan and sweep scan are available

Step scan:

- Probe pauses at each point to collect data
- Multiple samples averaged at a single point
- Lower noise measurement, with reduced solution stirring



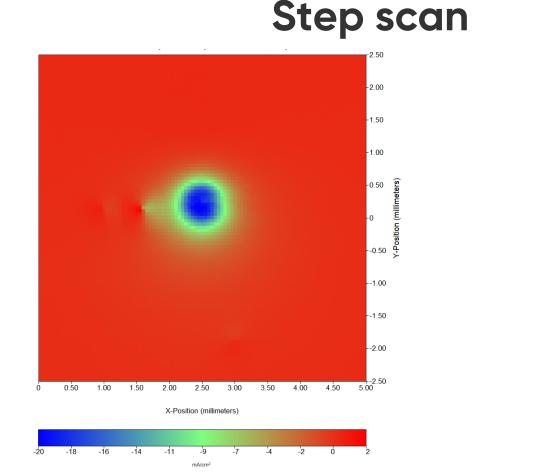
Sweep scan:

- Probe does not stop during a line, measuring at given time intervals
- Single sample measured at each point
- Faster measurement

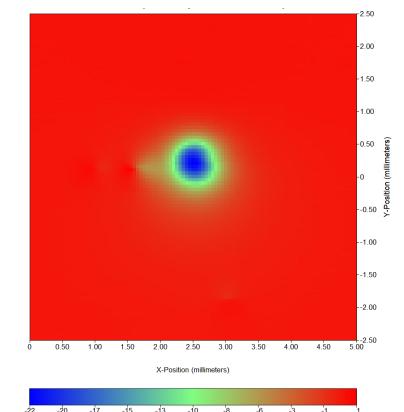


Step scan vs sweep scan example.

Step scan typically results in higher quality images than sweep scan. However a step scan experiment will take longer to perform.





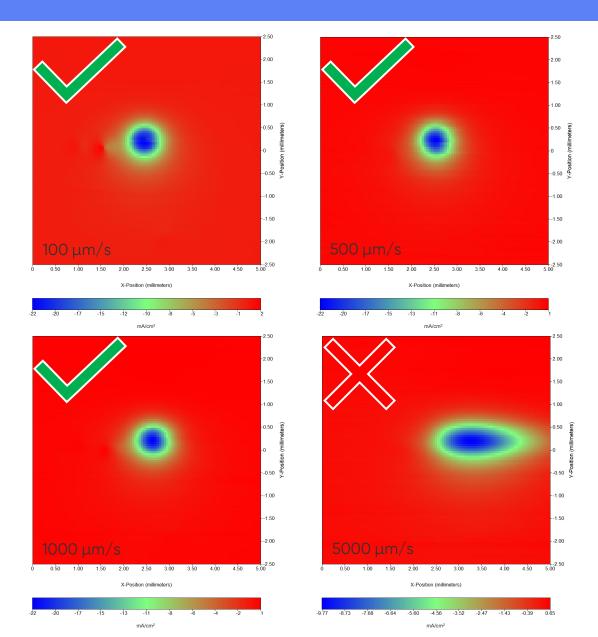


PIS in tap water biased at $-7.5 \,\mu\text{A}$

Selecting positioning scan rate.

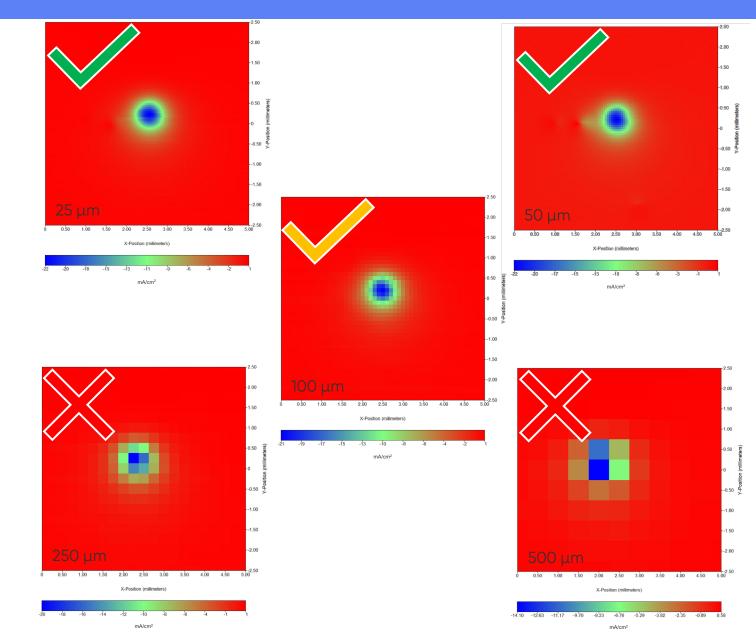
Positioning scan rate selected based on:

- Maintaining positioning accuracy
- Avoiding excess noise
- Avoiding signal distortion
- Reducing experiment time



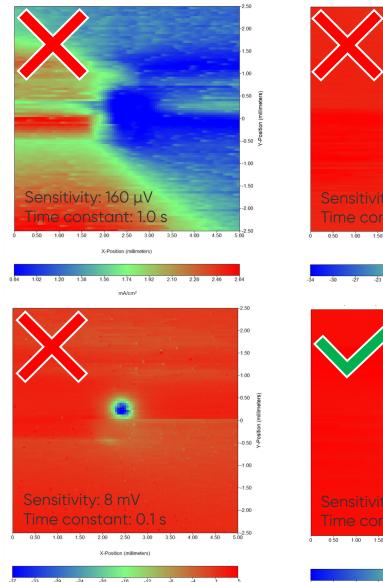
Selecting a step size.

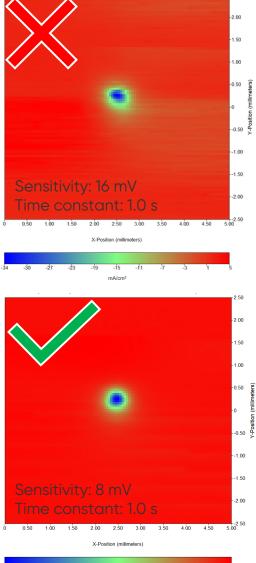
- While resolution is ultimately dependent on probe size, step size also matters
- Oversampling, where the step size is smaller than the probe, is common
- Smaller step sizes lead to clearer images but increase experiment times



Amplifier settings.

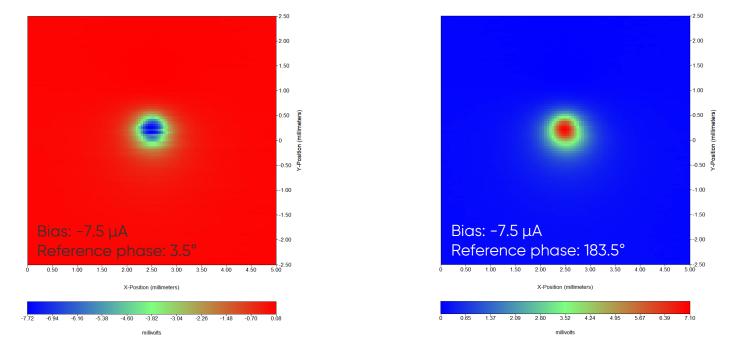
- The full scale sensitivity must be large enough to avoid signal overload, but small enough to avoid excess noise.
- The output time constant is selected in relation to the measurement speed. 1.0 is used for slower scans, 0.1 for faster scans.





Reference phase

- The sign of the raw voltage signal measured in SVET is dependent on the reference phase used
- When the reference phase is changed by 180° the signals will be of equal but opposite magnitude



Note: For clarity the raw SVET data is shown

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A number of considerations have been discussed with the aim of optimising the SVET measurement. These considerations relate to the sample, electrolyte, and configuration settings. Understanding how to control each of these settings will allow users to measure standard and novel samples.



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Thank you