BioLogic

How to get clear images in Scanning Droplet Cell (SDC)*

Version 1.0

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

Aim.

This tutorial aims to provide users with the information they need to obtain clear images using Scanning Droplet Cell (SDC, also known as Scanning Droplet System; SDS). We will address a number of factors which can affect the SDS 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.

Sample requirements.

- **In an SDC experiment the sample is the working electrode in the droplet cell**

- **An electrical connection is required**

- **Ideally the sample should be smooth and level**
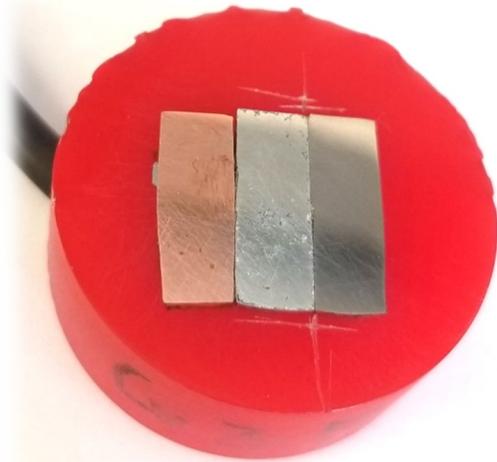
Mounting the sample.

Blu-Tac

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

Beeswax

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

Epoxy Resin

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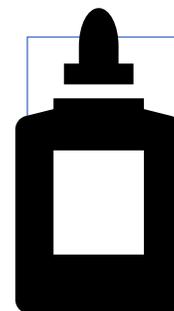
Other

PTFE tape, parafilm, double sided tape, glue, adhesive medical tapes...

Making electrical contact.

In SDC an electrical contact to the sample is required for it to be used as the working electrode in the droplet cell

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.

The SDC head.

The SDS470 is provided with two different SDC head types:

• Flow head
- Has a 500 μm aperture
- Electrolyte refreshed throughout experiment
- Used for studies of the effect of flow rate
- Used for studies of processes producing product
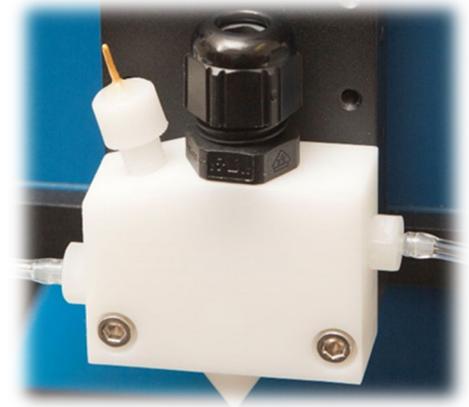
• Reservoir head
- Has a 100 μm aperture
- Electrolyte is the same throughout experiment
- Used when the highest resolution is needed
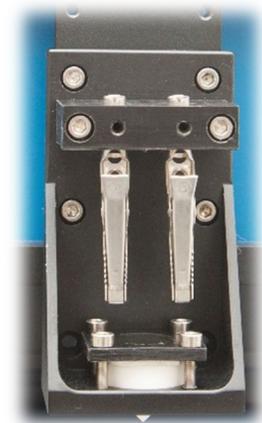
The diameter of the SDC head affects the lateral resolution

SDC Flow Head
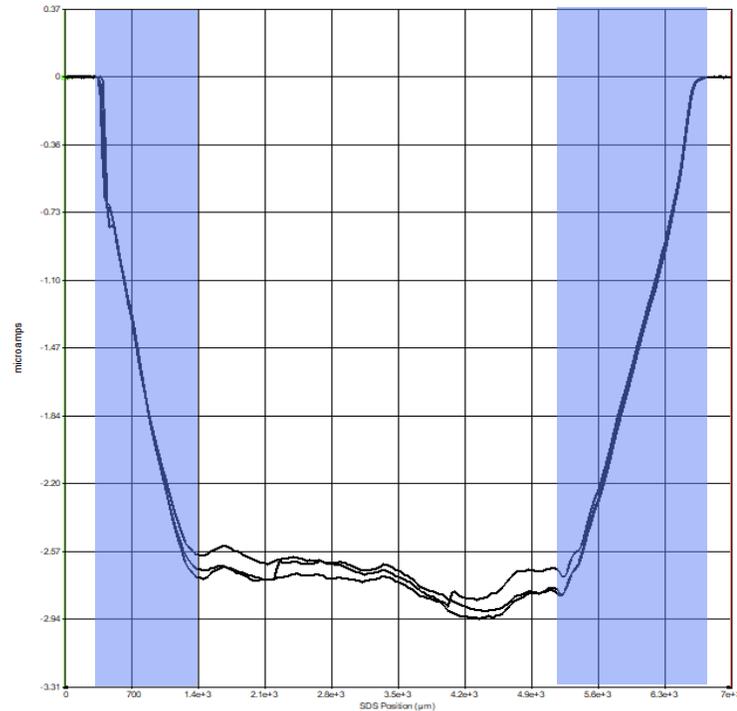
SDC Reservoir Head

Sample biased at –1.25 V *vs* Ag/AgCl
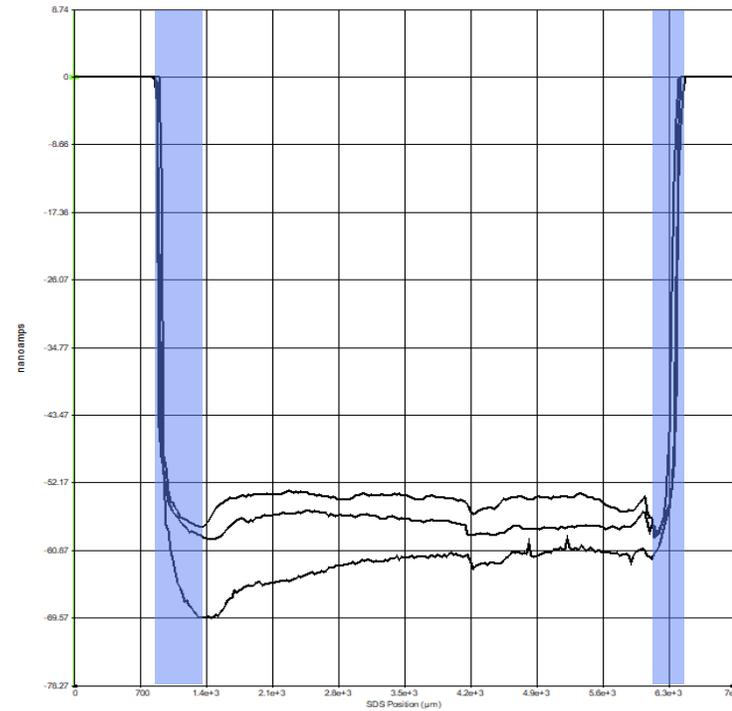
Sample biased at –1.25 V *vs* Pt

Standard 5 mm Au disc sample measured with tap water droplet for comparison

The transition region is sharper for the reservoir head which has a smaller aperture size.

Sample tilt and topography.

- **To maintain the droplet the SDS head should be near the sample surface (~100 µm)**
- **It should not touch the surface at any point in the scan**
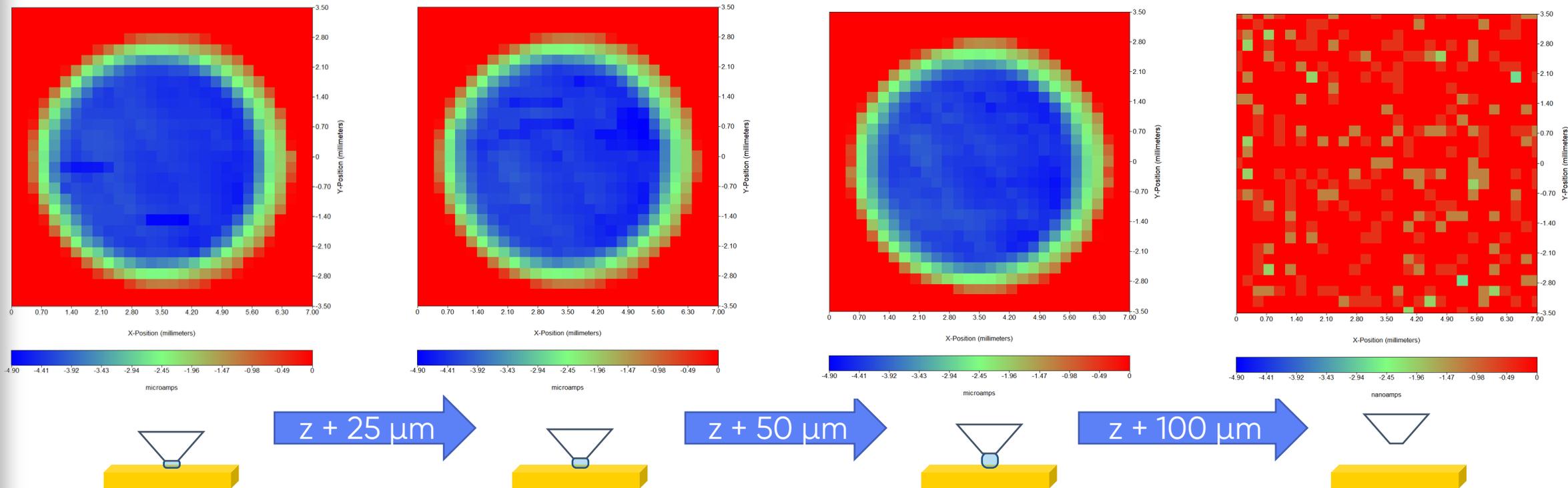
z + 25 µm   z + 50 µm   z + 100 µm

Sample: Au biased at –1.25 V *vs* Ag/AgCl. Electrolyte: Tap water

- **Too much sample tilt causes partial or complete loss of sample-droplet contact**

- **Too much sample tilt can cause a loss of signal**

- **Sample tilt can lead to probe crash**
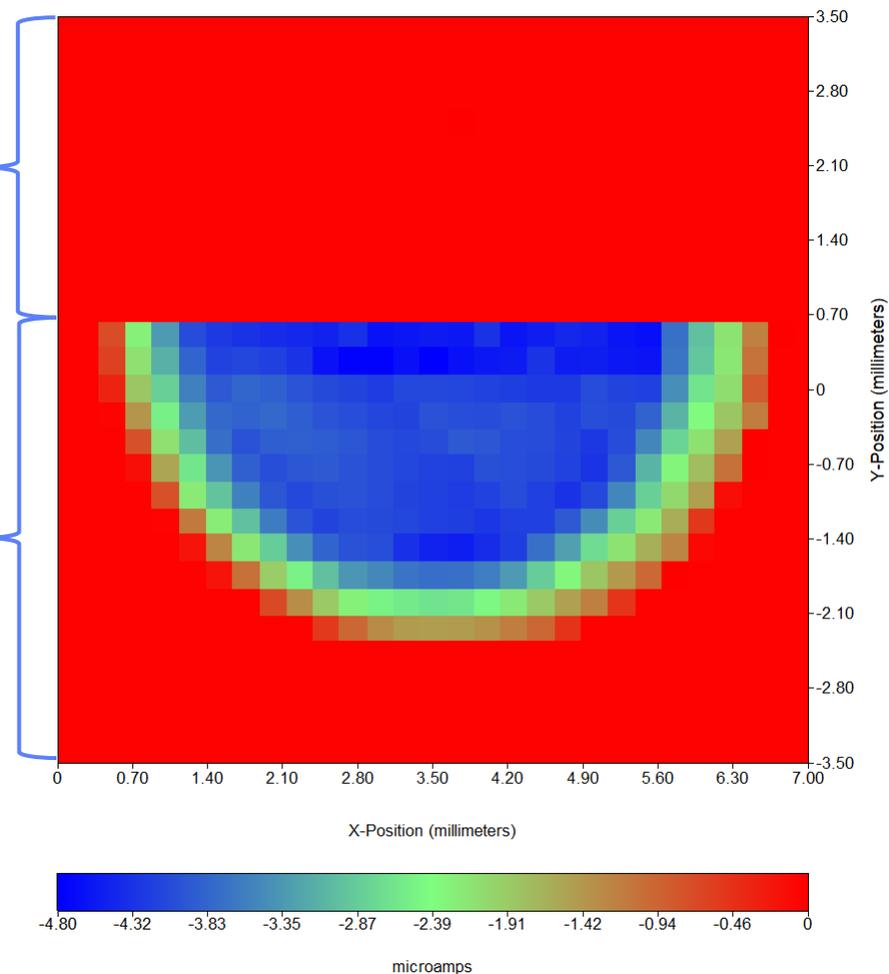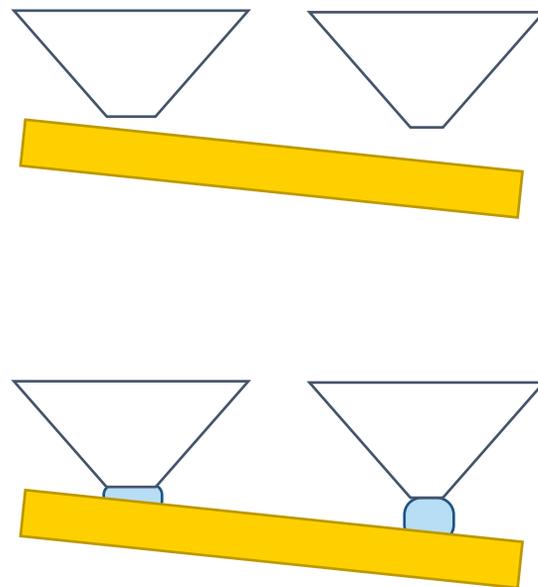
Sample: Au biased at –1.25 V *vs* Ag/AgCl.

Electrolyte: Tap water

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

Levelling with adjusting screws

Configuration settings.

Step scan or sweep scan.

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

Step scan:

- **Probe pauses at each point to collect data**
- **Multiple samples measured at a single point**
- **Samples averaged**
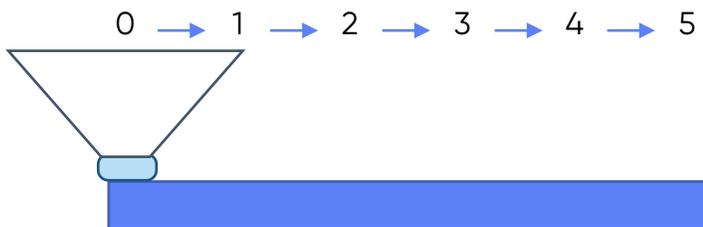
Sweep scan:

- **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
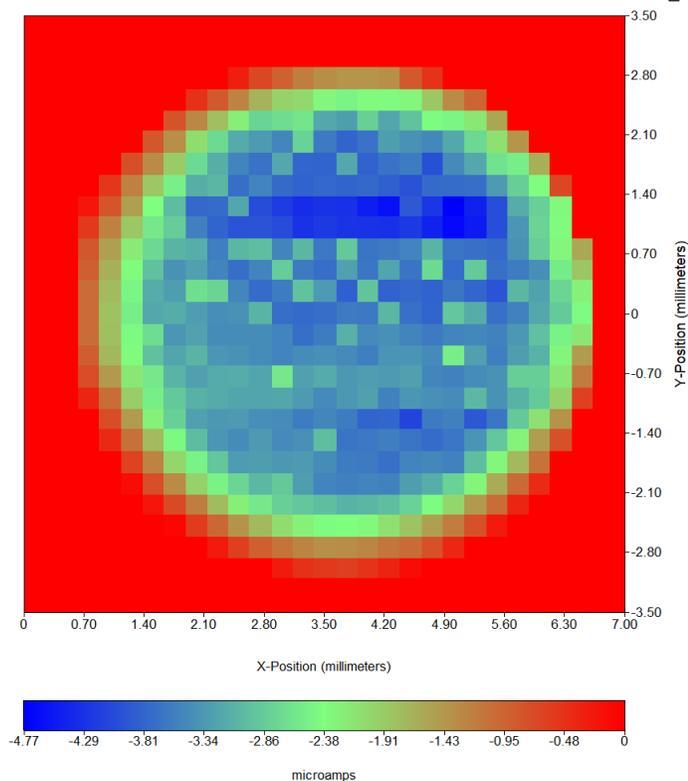
Sweep scan
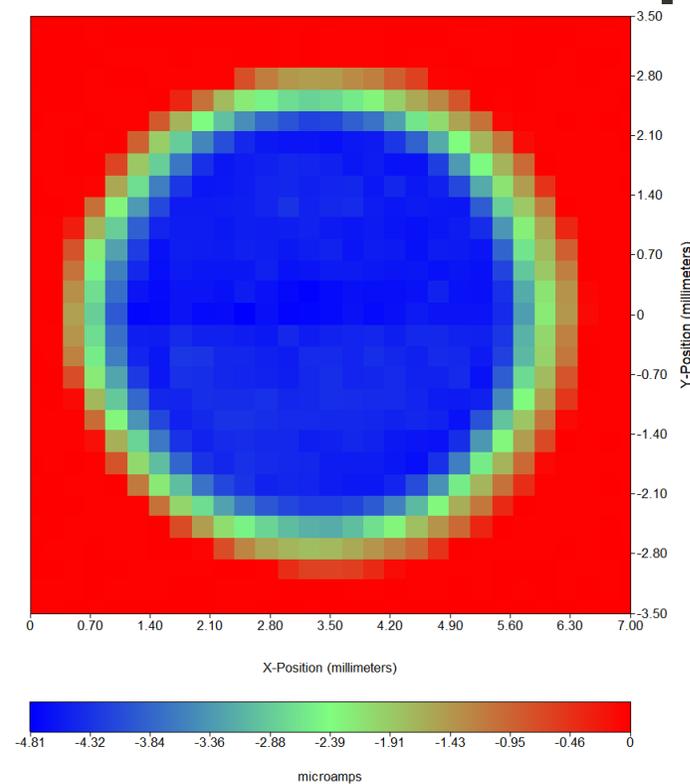
Sample: Au biased at –1.25 V *vs* Ag/AgCl. Electrolyte: Tap water

In step scan it can be difficult to overcome noise added to the system from the peristaltic pump. Reducing the flow rate and sampling rate can help.

Selecting positioning scan rate.

Positioning scan rate chosen to:

- **Maintain positioning accuracy**
- **Avoid excess noise**
- **Avoid signal distortion**
- **Reduce experiment time**
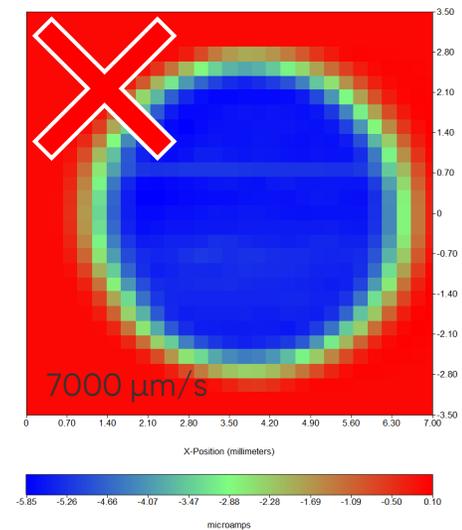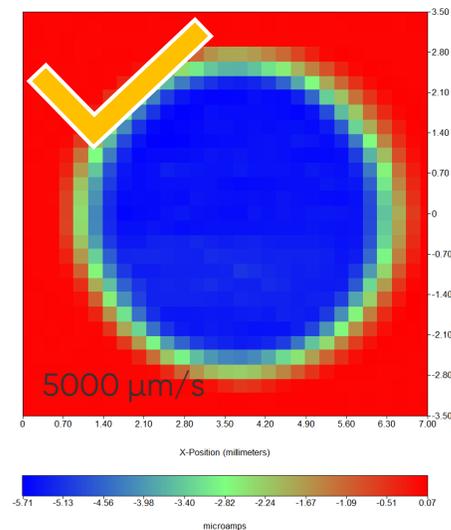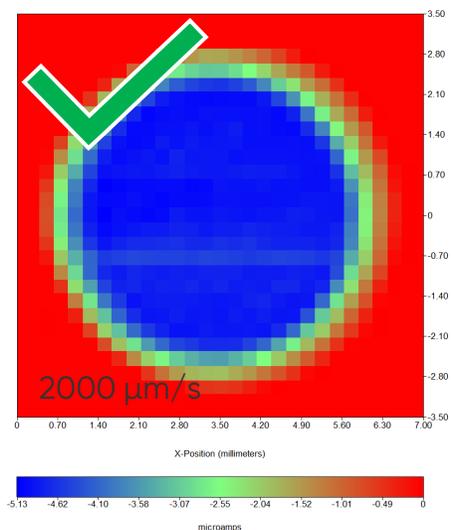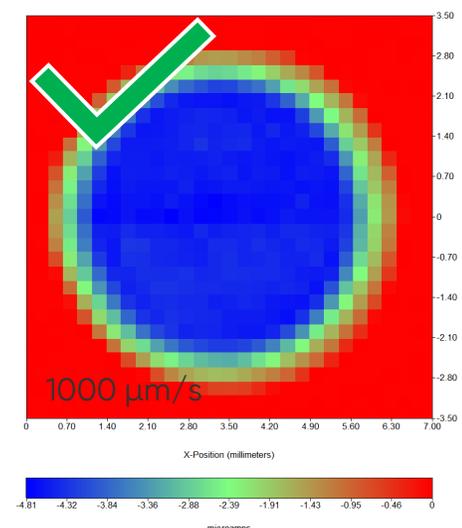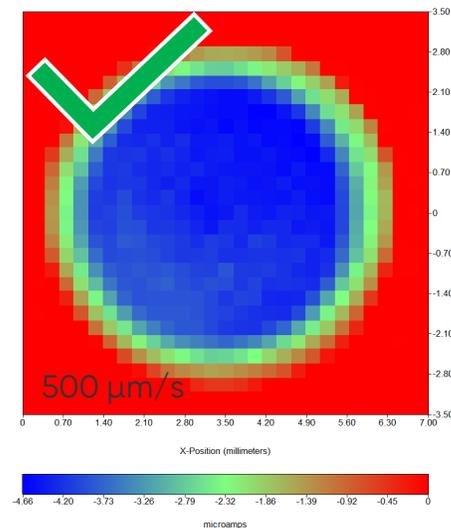

Sample: Au biased at –1.25 V *vs* Ag/AgCl. Electrolyte: Tap water

- **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**
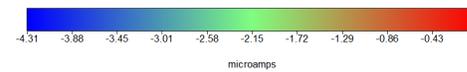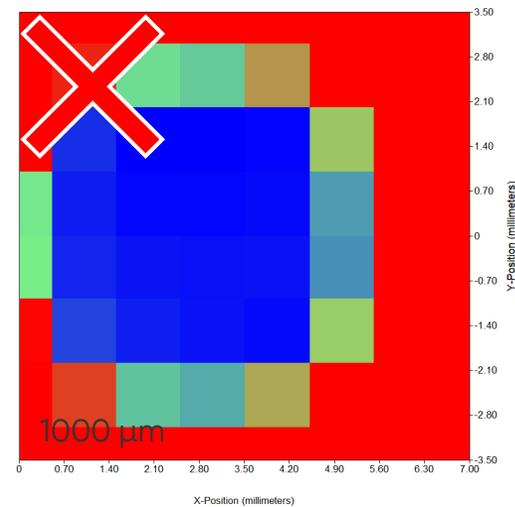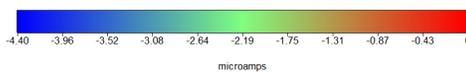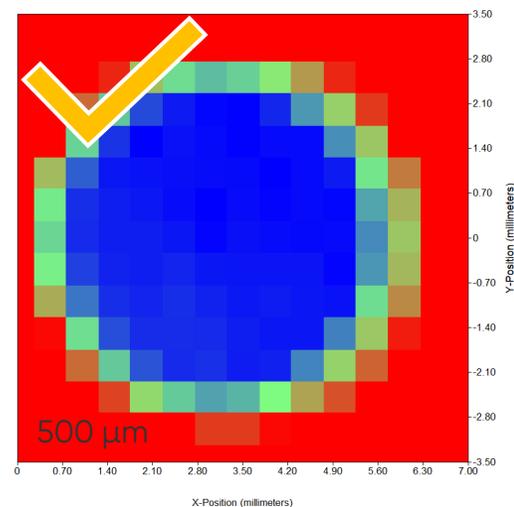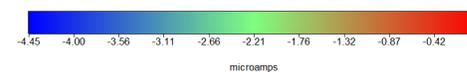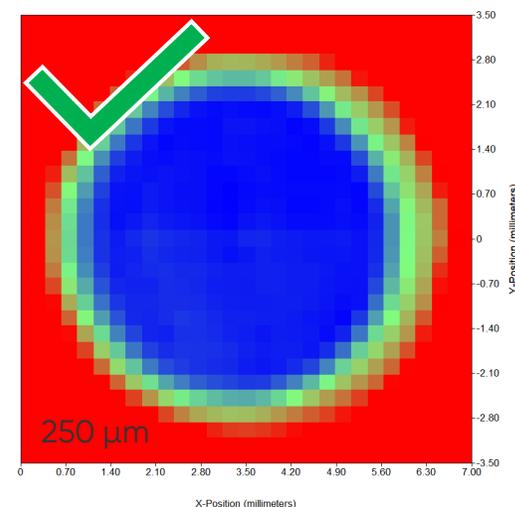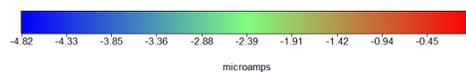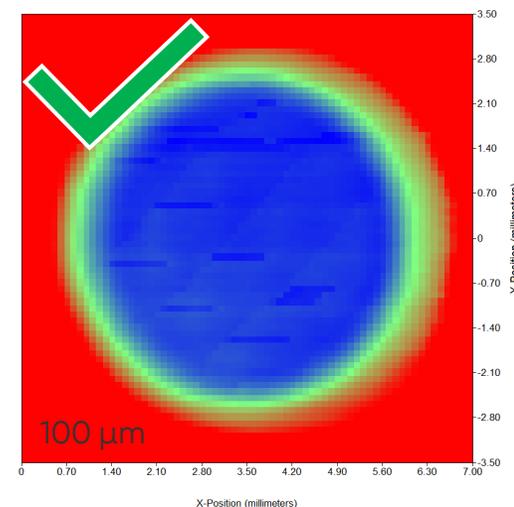
Sample: Au biased at –1.25 V *vs* Ag/AgCl.

Electrolyte: Tap water

Flow out vs. flow in.

- **Flow out should be faster than flow in**

- **The droplet grows on the trailing side if flow out : flow in is too small, stretching features**

- **Air enters the head if flow out : flow in is too large**

- **If scan rate changes flow out : flow in should be updated**
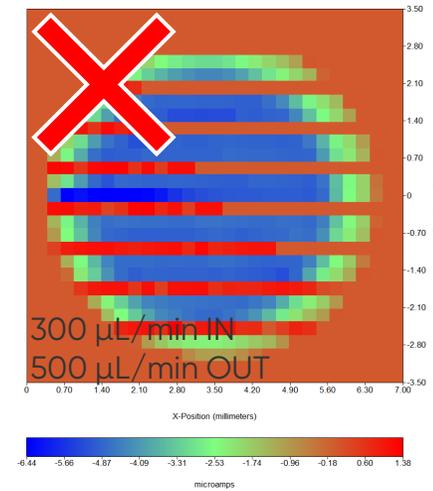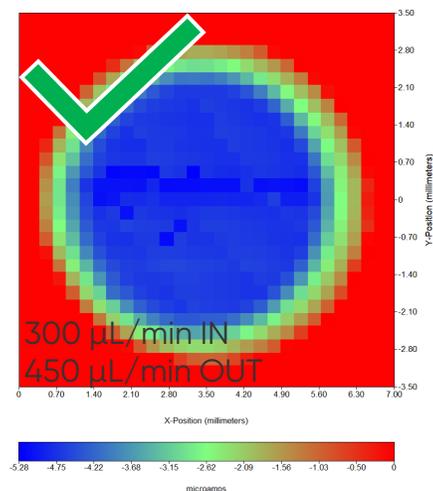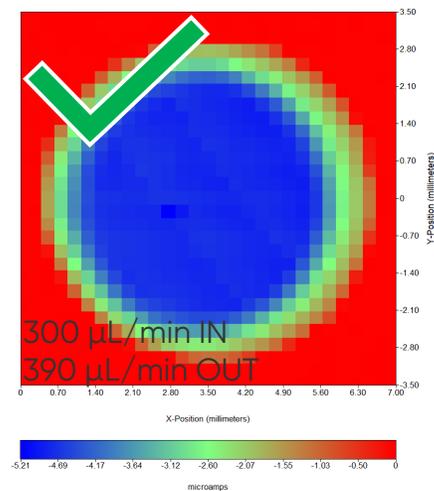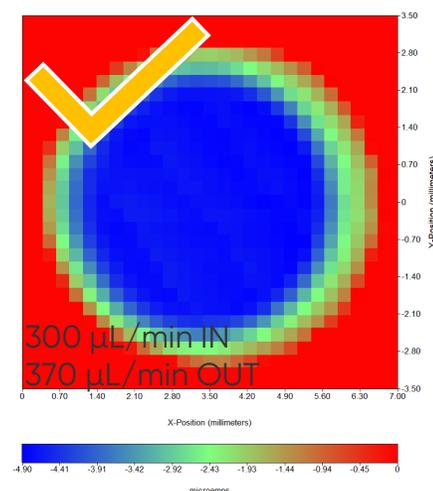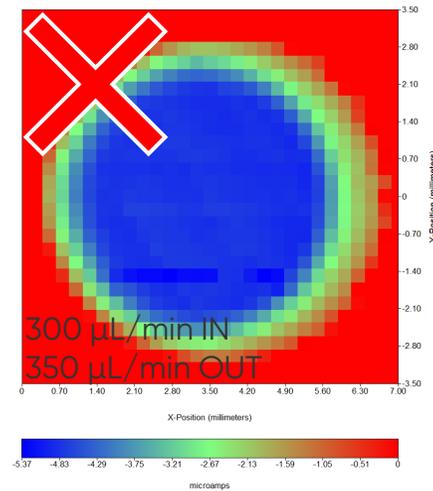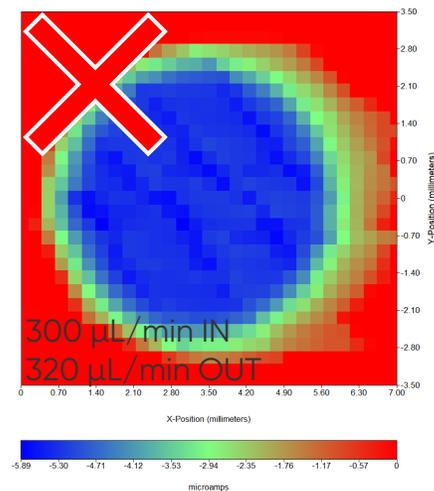
Sample: Au biased at –1.25 V *vs* Ag/AgCl. Electrolyte: Tap water

What happens if there is a bubble?

- **If there is a bubble present the three electrode cell breaks**

- **As the droplet passes the RE, or CE swings in the signal are seen**

- **See "Droplet balancing tips" slide**
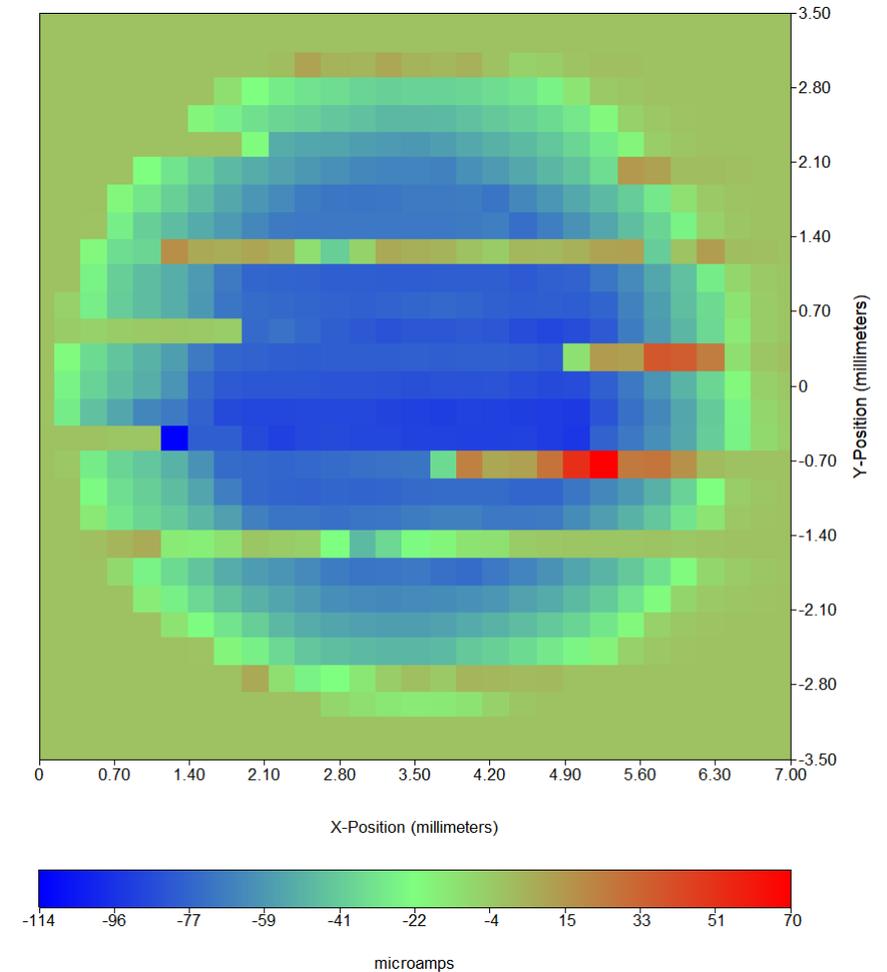
Sample: Au biased at –1.25 V *vs* Ag/AgCl.
Electrolyte: 100 mM KCl

Droplet balancing tips.

Sealing
All fittings should be wrapped in PTFE tape to ensure an airtight seal.

Flow rate
The flow rate in should be set slower than the flow rate out.

Distance
When the SDC head is positioned too far from the surface it can be difficult to balance the droplet.

Tubing
Old tubing may not be squeezed as well by the peristaltic pump.
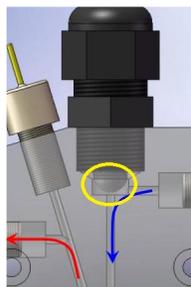
Reference Electrode
The position of the RE is important. Too low and it will block electrolyte flow, too high and it will not seal the head well.

Other SDC Experiment Options.

- **Local impedance measurement**
- **Sample does not need to be fully immersed**
- **Highly localized measurement**
- **Can be used to map impedance at a set frequency, or to measure local EIS spectra**
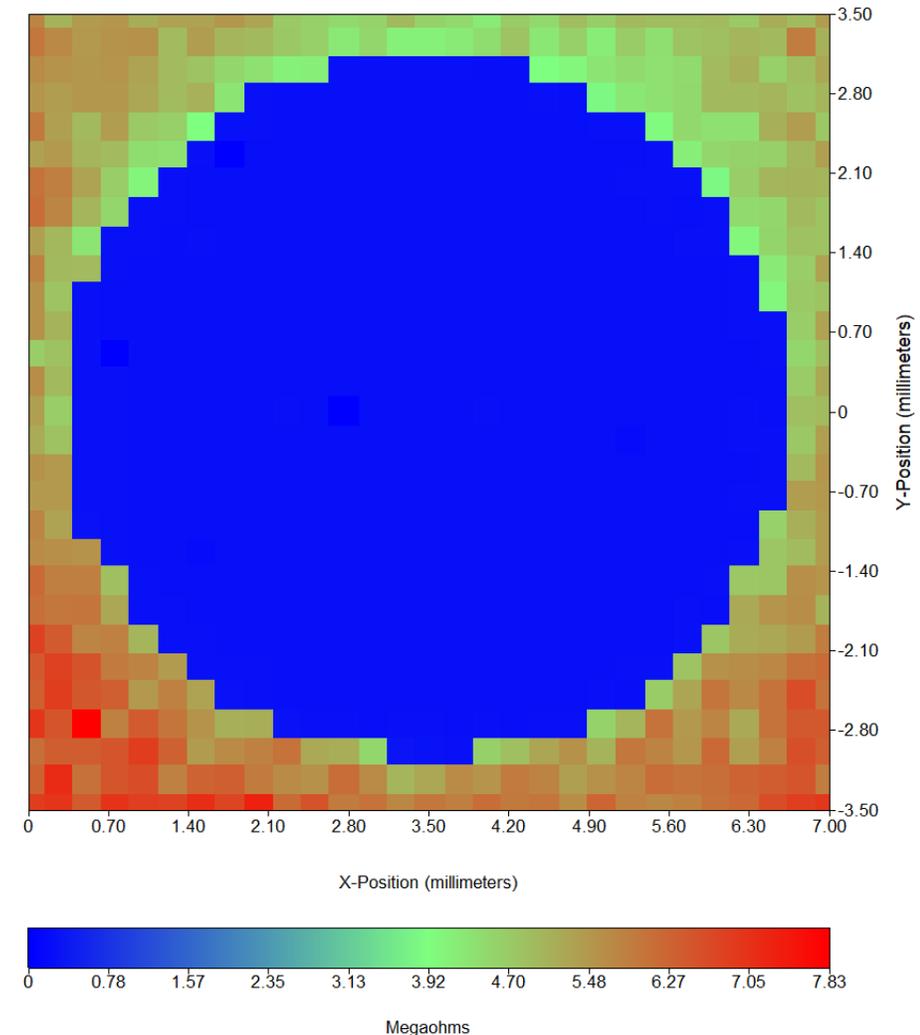
SDC for local electrochemical experiments.

- **Confining the electrochemical cell to a microdroplet makes SDC attractive for local electrochemistry experiments**

- **SDC can be used with all electrochemistry, corrosion and impedance measurements in the M470 software**

- **Local measurements can be performed with manual movement of SDC head between points or sequenced for automated movement and measurement**


Working
Electrode
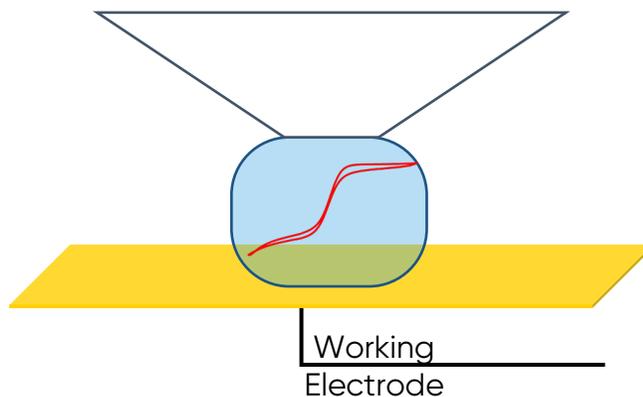
- **Experiment sequencer can be used to automate SDC local experiments**
- **Local experiments include cyclic voltammetry, and electrochemical impedance spectroscopy**
- **Logic steps like movement, and pump control, can be included**
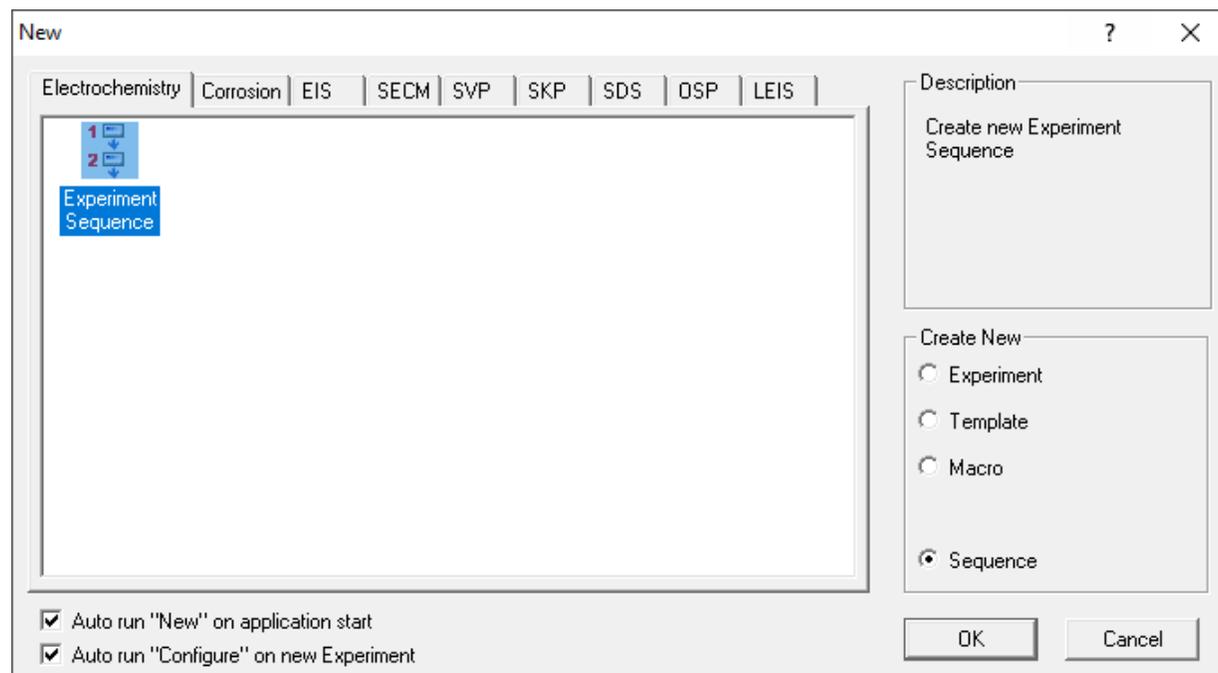
SDC sequence example.
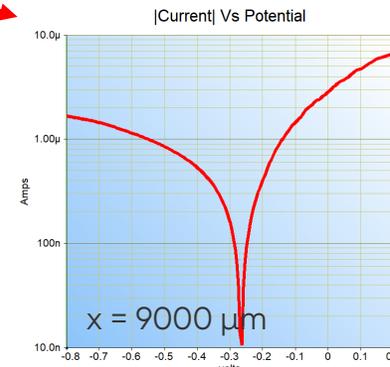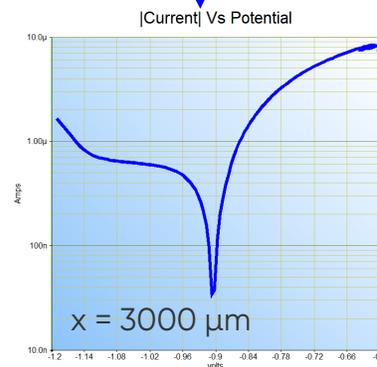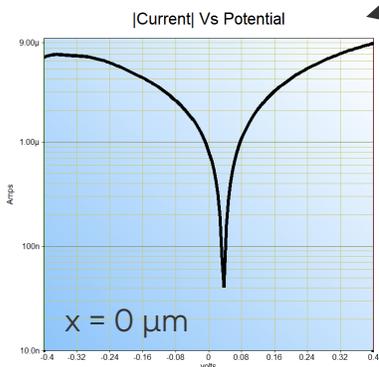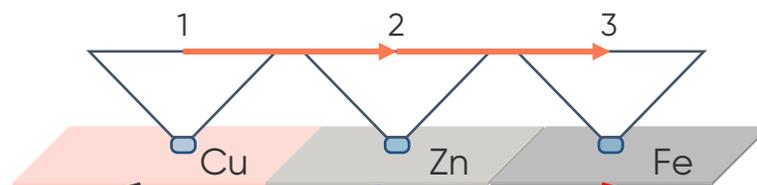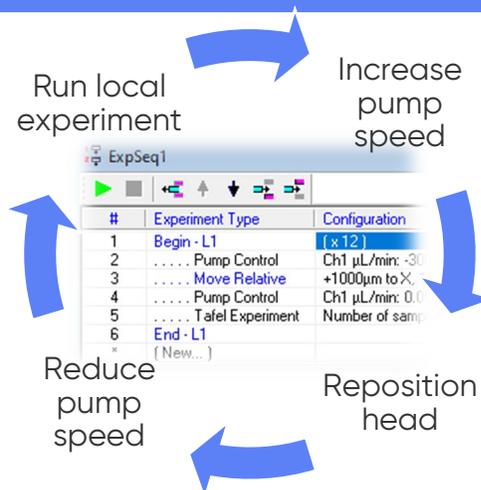
For more information see: SCAN-Lab TN#21

Conclusion.

Conclusion.

A number of considerations have been discussed with the aim of optimising the SDC 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.

More information.

BioLogic Support: https://www.biologic.net/support/

BioLogic Contact: https://www.biologic.net/contact/

BioLogic

www.biologic.net

contact@biologic.net

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Thank you!