

PeakForce SECM with Bio-Logic SP-300 Potentiostat

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Preliminaries:

1. To use a Bio-Logic SP-300 bipotentiostat in place of the CHI760E, follow the Bruker PeakForce SECM manual for all settings except for the CHI760E bipotentiostat in the Bruker manual.
2. You must use the **ECLab Express** software for the Biologic bipotentiostat not the ECLab software.
3. Both ultra-low current probes (or amplifiers) on the end of the two SP-300 cables **MUST** be inside the AFM hood as shown in Figure 1, to get a noise level of ~ 10 pA at 10 nA current range with a 1 kHz filter. The AFM hood acts as a Faraday cage for the experiment.

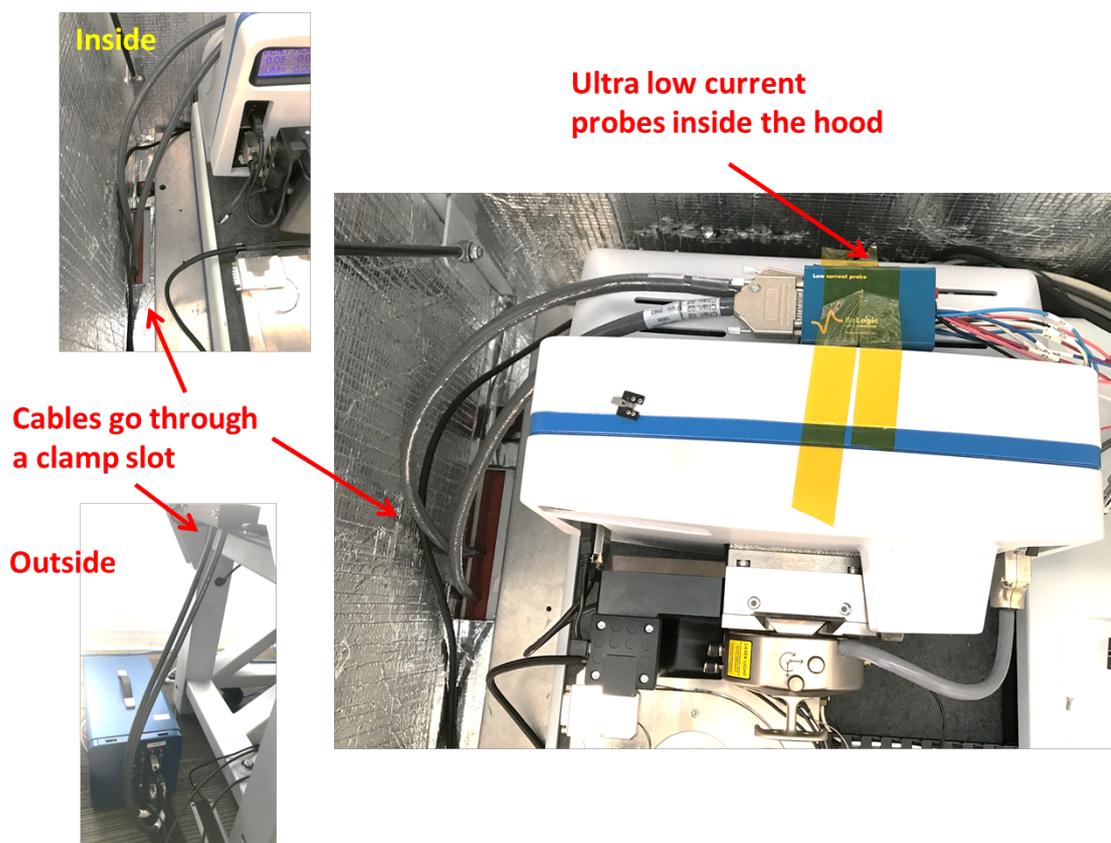


Figure 1. Low Current Probes (or Amplifiers) located inside the AFM hood, an effective Faraday cage. Cables are routed through a sealed clamp slot.

- The bandwidth of the SP-300 bi-potentiostat when set to a current range of <1 nA is not known. A filter setting of 5 Hz is too slow for AFM imaging. From our testing, 1 kHz and 50 kHz have similar noise levels. This may suggest that the bandwidth might be below 1 kHz.
- The noise level was tested using CA setting of 0.03 s/point, a filter of 1kHz, and a current range at 10 nA. The noise has a range of $\sim \pm 10$ pA or 20 pA peak-to-peak and a standard deviation of ~ 9 pA, Figure 2.

Installation:

- Connect the two current cables(at least one must be the ultra-low current cable for the tip electrode) to the SP-300 Potentiostat and insert the two cables into the hood of the Dimension Icon through the holes at its bottom, Figure

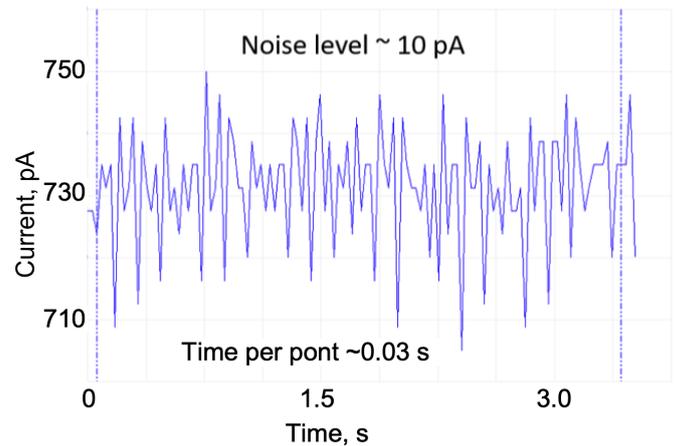


Figure 2. Typical current noise level (~ 10 pA) in CA mode. The noise level was tested using CA setting with setting time interval at 0.03 s, the filter at 1kHz, and the current range at 10 nA.

- Channel-1 and -2 cables are shown on the left and right; respectively, Figure
- The potentiostat cables can be fixed to appropriate position using tape, Figure
- Low-current probes (blue ports) should always be inside the AFM Hood

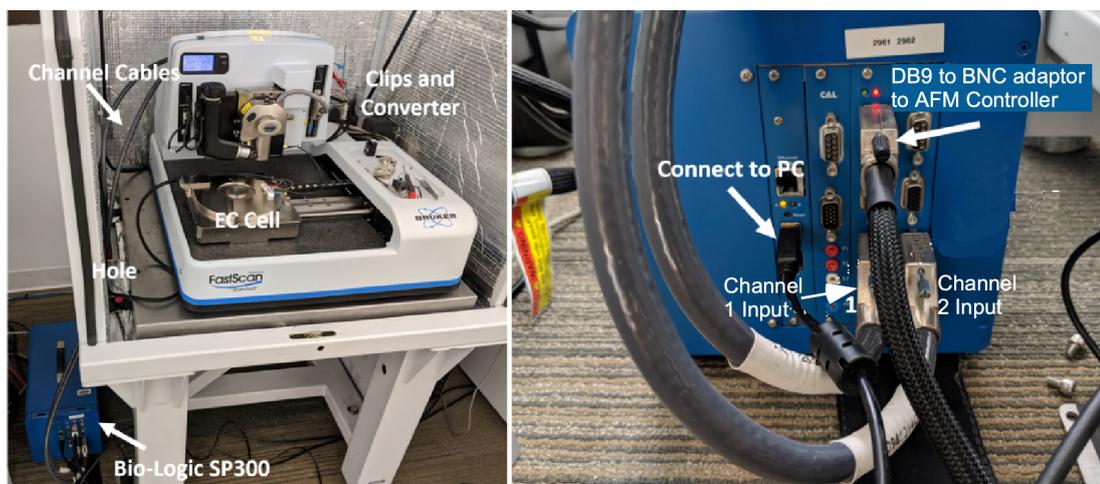


Figure 3. Hardware configuration between the Bio-Logic SP-300 potentiostat and the Dimension Icon AFM

- during measurement to reduce the noise level of the gained current signal; (Figure 3)
2. For the use of the Bio-Logic as a bipotentiostat you **must** configure the instrument to use the counter electrode (CE) as ground (CE to Ground) configuration. To do this:
 - a) In the **ECLab Express** software under Hardware configuration choose CE to Ground or in “Advanced Settings”, select the “CE-to-Ground” option, Figure 4
 - b) **Disconnect the cables from the cell.**
 - c) Connect the P1 and S1 (red) leads to the working electrode, Figure 4.
 - d) Connect the S2 (black) lead to the reference electrode
 - e) Connect the GROUND (black) and S3 (blue) leads to the counter electrode
 - f) The P2 dark blue cable is not used (open-circuit).
 - g) Using the alligator clips, these cables can be connected to the multi-channel BNC converters provided by Bruker and finally to the small electrochemical spring terminal on the SECM chuck, Figure 5;
 3. The potentiostat cannot auto range you must choose a range to use.
 4. The output of the SP-300 Bio-Logic can be connected to the AFM controller by use of the female DB9 connectors on the SP-300, Figure 3. Use the Bio-Logic DB9 to multi-BNC converter. Two of the outputs from the converter are labeled E and I. Note that the E and I outputs have the opposite polarity to the measured values i.e., if the analog output -2V, it is actually +2V at the cell (see ECLab Express Installation and Configuration manual).
 5. For SECM experiments, connect the Bio-Logic channel-1 S1 and P1 to the nano-tip working electrode and channel-2 (S1 and P1) to the sample.
 6. Connect Channel-1 outputs E and I to the AFM controller using the “Low

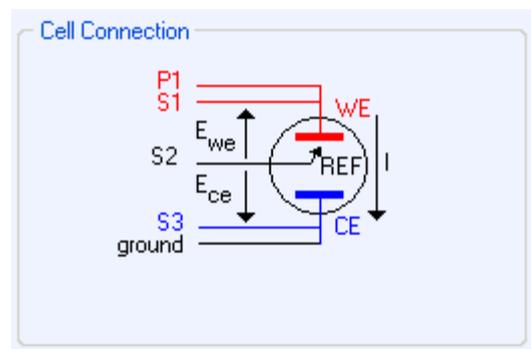


Figure 4. CE to ground connection mode for using SP-300 as bi-potentiostat.

Frequency Analog” panel inputs 1 and 2, respectively, Figure 6.

7. To monitor the sample current (or working electrode 2 in a 4-electrode configuration), connect Channel-2 output I to Lock-In 3 input, Figure 6.

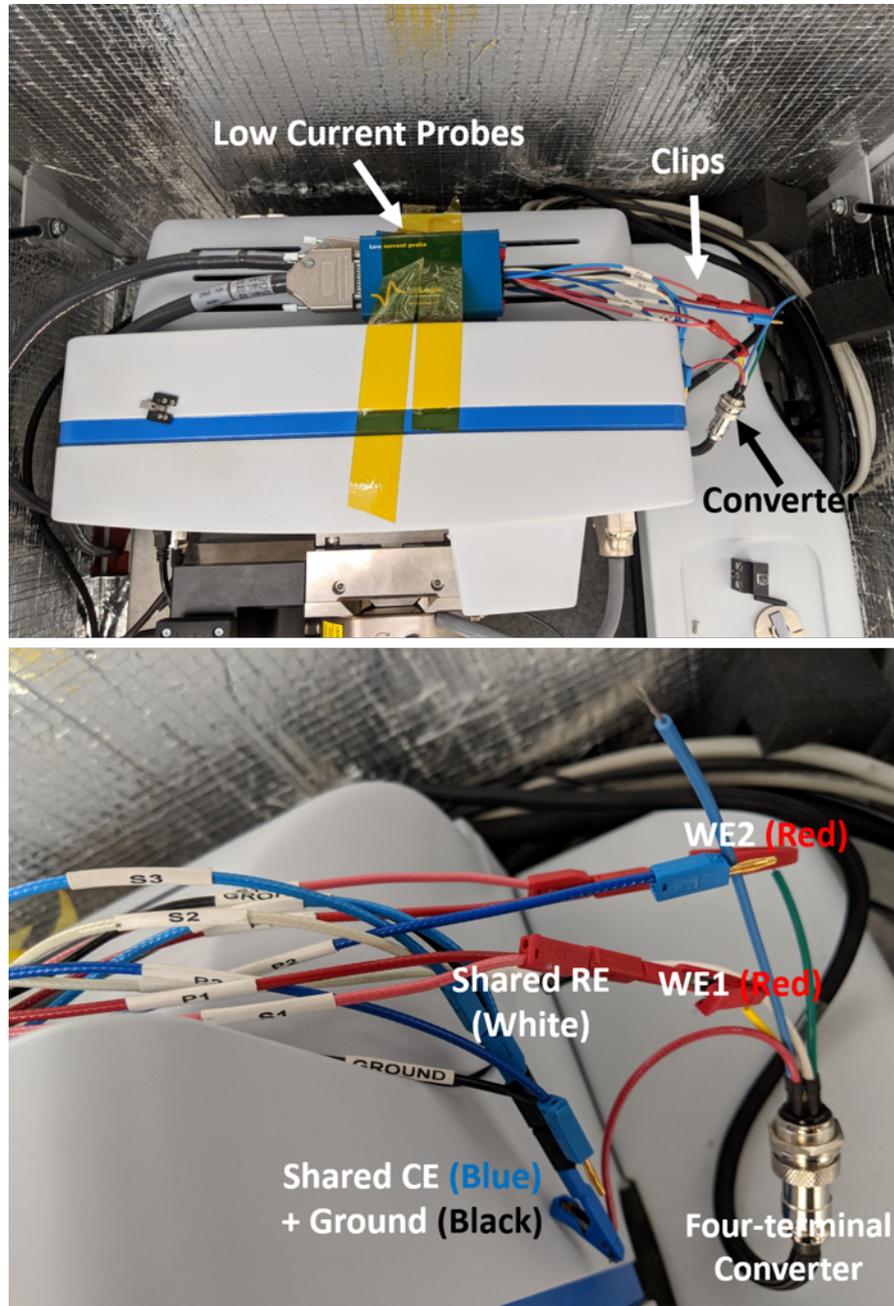


Figure 5. The connection of Bio-Logic Cables and Clips and the four-terminal converter inside the hood of the Dimension Icon

8. Before starting EC experiments, in the **ECLab Express** “Advanced Settings”, make sure to select the “CE-to-Ground” option chosen for a bi-potentiostat, Figure 7. The band width can be set at 1kHz to allow data collections consistent with scanning speed of the tip. In selecting an experiment, one can either choose a single-channel experiment (CA/CV) that will only monitor Channel-1, or a bi-potentiostat experiments (CV-CA, CA-CA, CP-CA) by that monitors both Channels 1 and 2, Figure 8. Before starting an experiment, a dialog box may appear asking you to the change the electrode configuration, be sure to select “No” and keep the configuration at “CE to ground”, Figure 7-8.

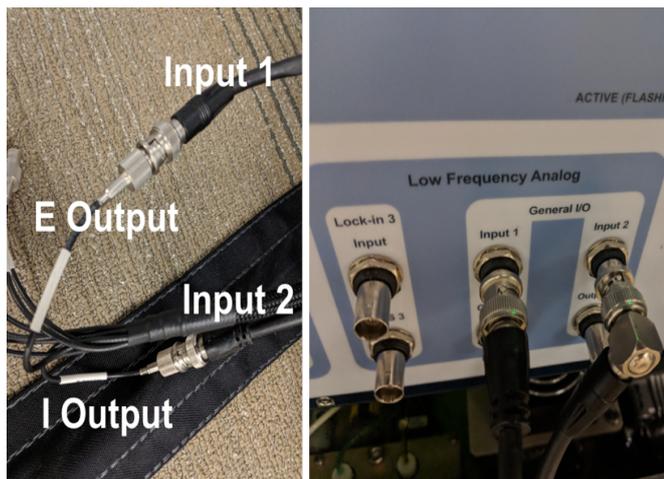


Figure 6. The BNC connection from the SP-300 potentiostat (E/I signal) to the AFM controller (Input 1/2) so that the electrochemical signals can be directly recorded on the NanoScope Software. To monitor the sample current (or working electrode 2 in a 4-electrode configuration), connect Channel-2 to Lock-In 3 input.

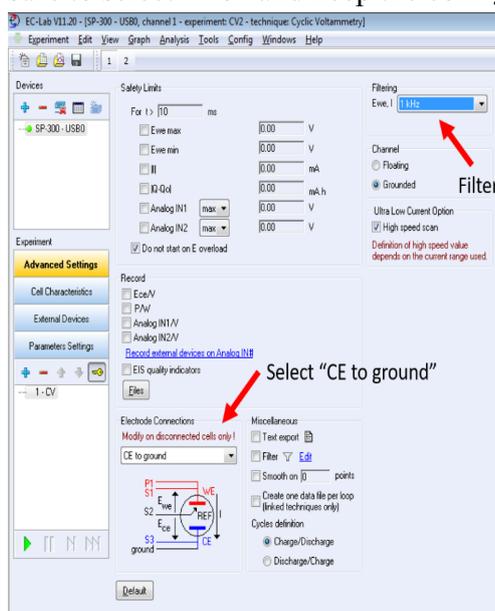


Figure 7. Selection of the electrode connection and the filtering band width in the ECLab Express software

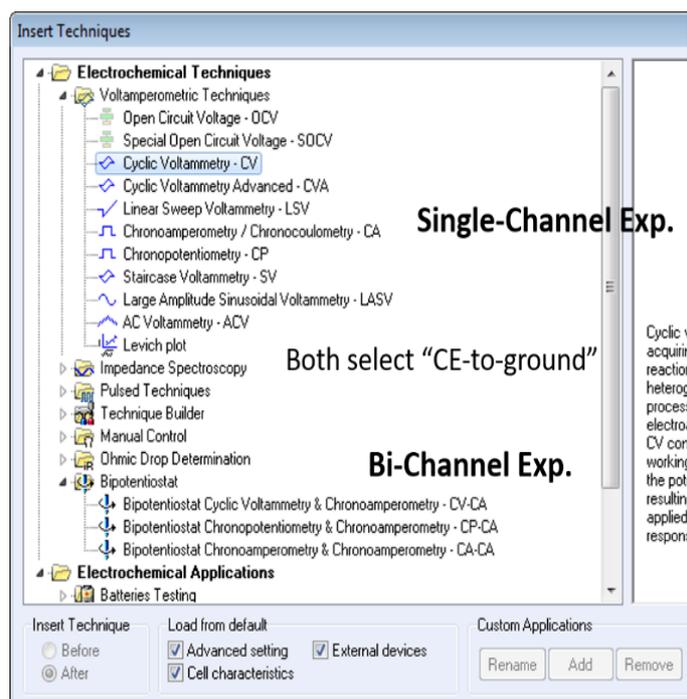
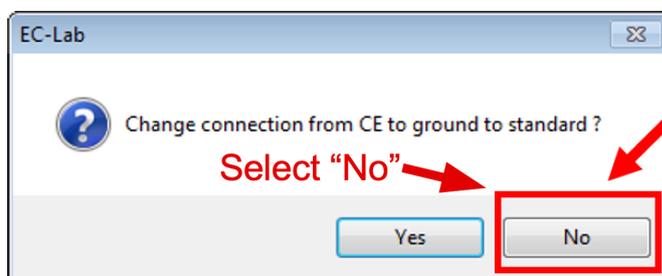


Figure 8. When selecting either a single-channel or a bi-channel experiment, be sure to select “CE to ground” as the electrode connection



9. During the scan, the noise level in CA mode with a time interval of 0.03 s (30 ms/data point) and band width of 1kHz was found to be ~10 pA (Figure 2).
10. When recording electrochemical data in the NanoScope Software using the XY data capture, the potentiostat and the ECLab Express and the NanoScope software must be set to the same range NanoScope Software. For example, if the I-range is “1 nA” in the ECLab Express, “1 nA/V” should be selected in the NanoScope Software.
11. Figure 9 shows a typical CV graph of the tip working electrode in the NanoScope Software recorded by the AFM controller. Note that the polarity of the gained

current data is reversed in the NanoScope software (see Part 3).

12. **[Warning]** During experiment, an obvious discharging behavior from the tip

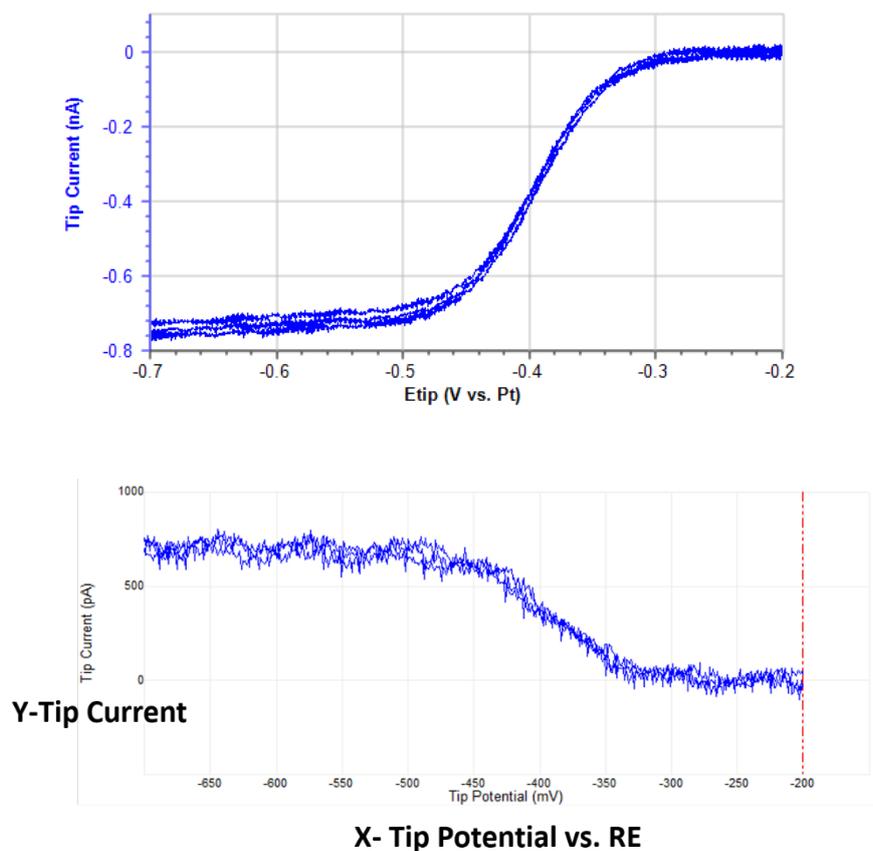


Figure 9. Typical CV curve of the tip electrode recorded in the Bio-Logic software and in the strip chart of the NanoScope software. Note that the polarity is reversed which can be adjusted in the NanoScope software

electrode at the beginning of each experiment was observed. This may be due to the ultra-small size the tip. This discharge may impact the quality of the electrochemical data gained at the start of experiment. For example, The Figure 10 shows several continuous CV curves of the tip electrode. Due to the discharging behavior, the first CV was impacted and the CVs obtained later on appeared more well-defined and reasonable.

13. From here on follow the PeakForce SECM manual for your experiment. Below is a screen show of SECM tip current profiles at different tip-sample distance. The line plots on the right from bottom are at nominal 30, 100, 200, 400, and 750 nm tip-sample distance. The noise level is about 10pA (std = 9.3 pA).

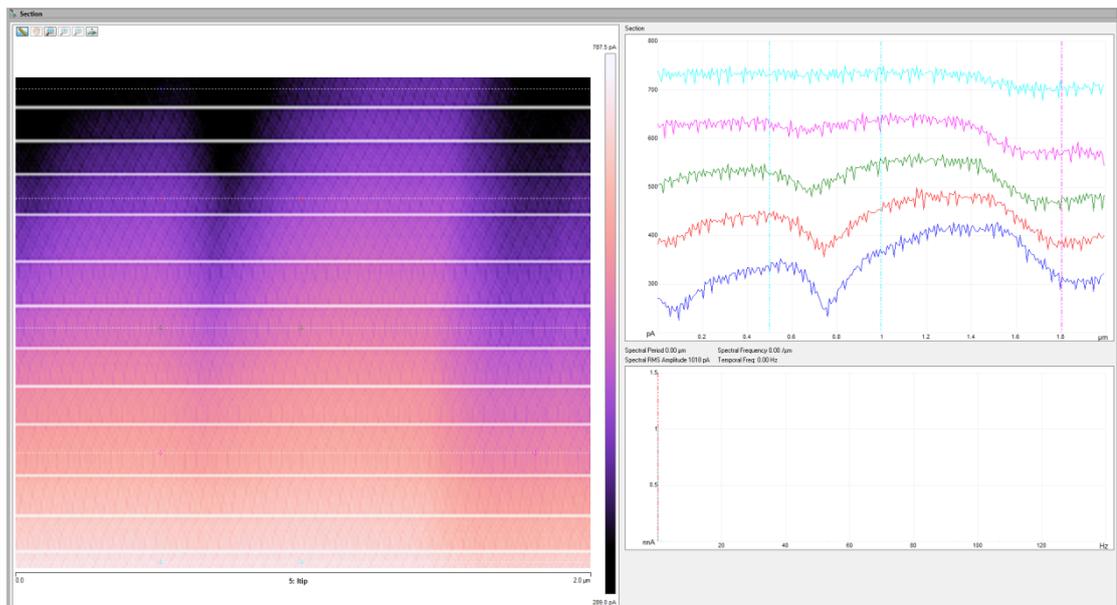


Figure 11. A typical SECM results using the Bio-Logic SP-300 bi-potentiostat.

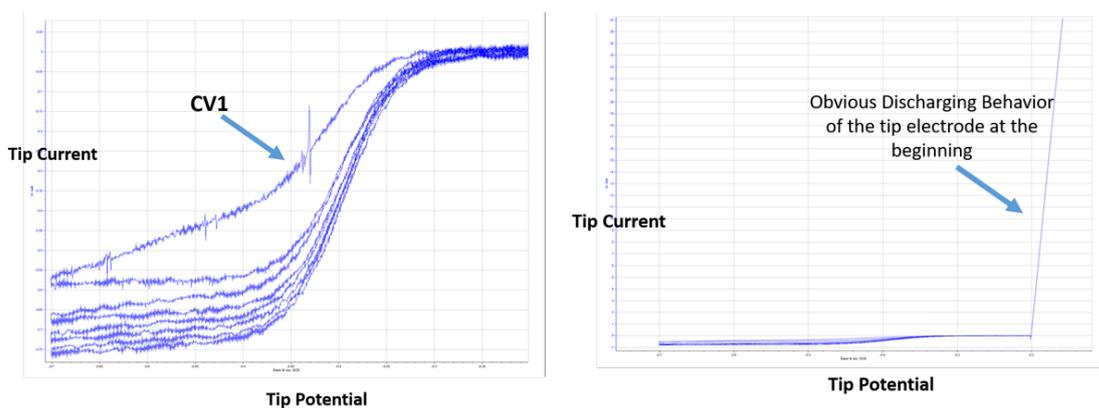


Figure 10. Typical charging behavior of the tip electrode at the beginning of experiment and its impact on the gained first CV curve