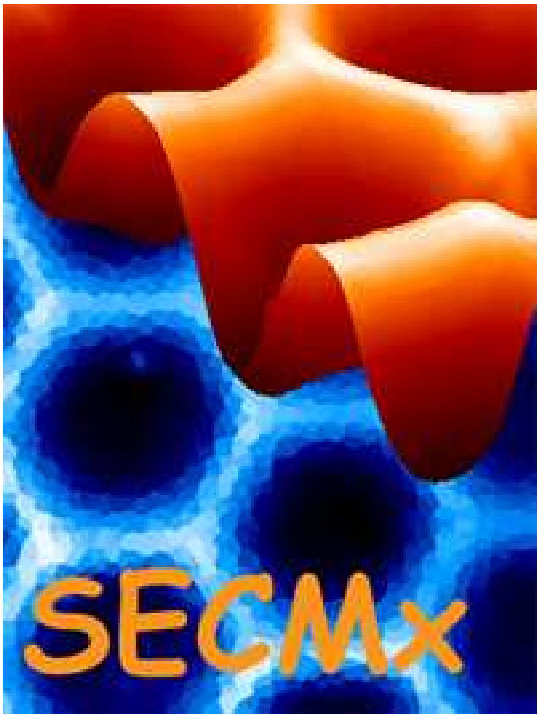


# Manual SECMx, version 14

# Part II: Operational procedures

Last change 10 August 2013

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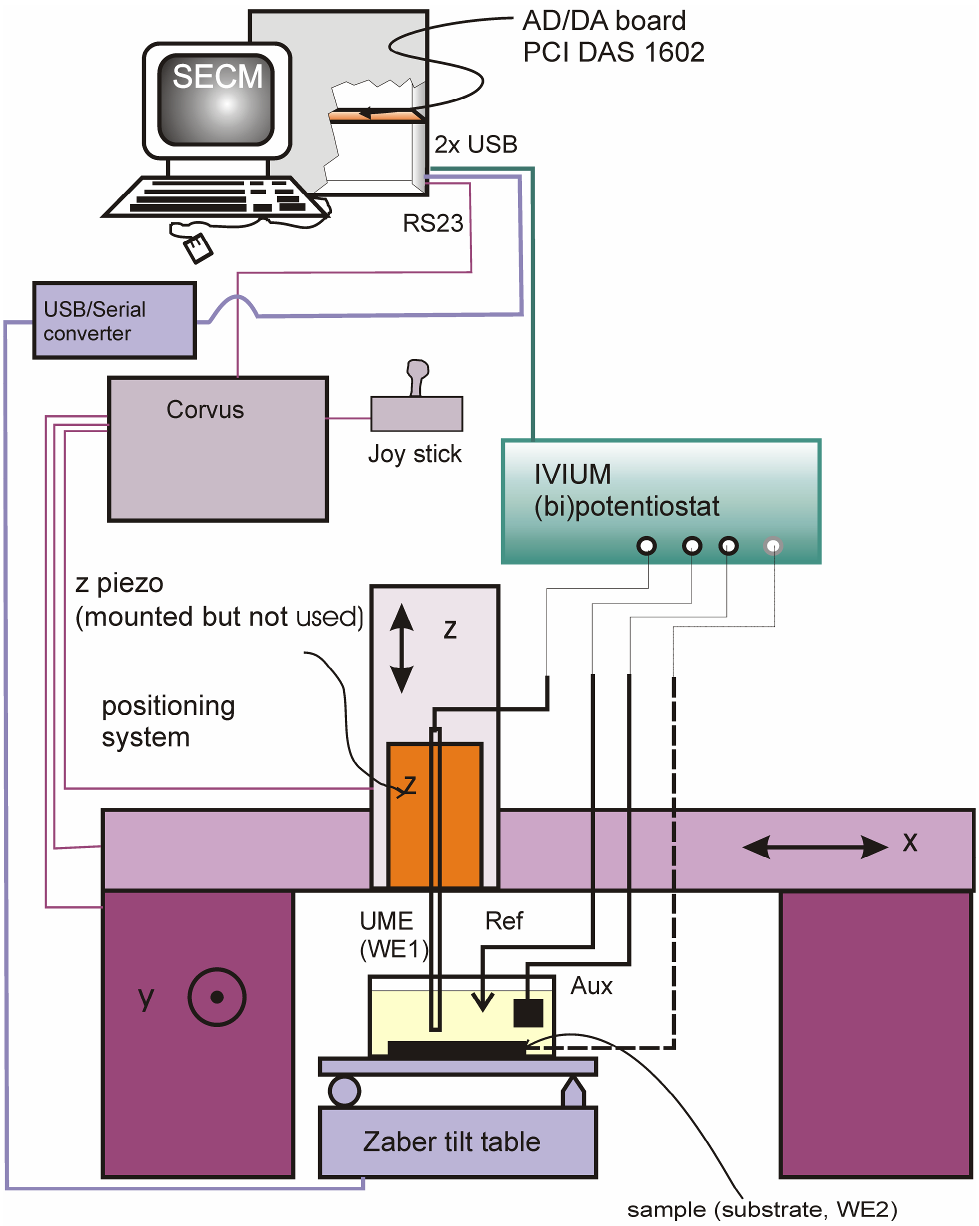
# 1 Standard setup (without using the shear-force distance control)

The hardware and software should first be installed as described in the instruction. The normal operation of the SECM is without the shear force option. It is strongly recommended that your first work without shear force. Once you are familiar with the SECM principles and the operation of this hardware you may advance to use the shear force. In order to setup the instrument, connect the instruments in the following way.

## 1.1 Using one UME as probe

### 1.1.1 Setup

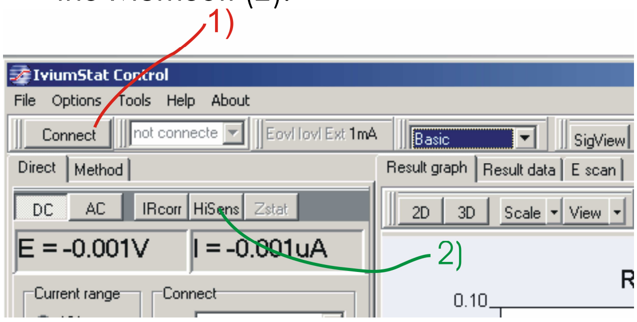
The setup is illustrated with a Märzhäuser positioning system, an Ivium Compactstat, and a Zaber tilt table. For the use of other positioning systems and potentiostats proceed in a similar way.



After connecting the microelectrode as WE1 to the potentiostat, fill the electrolyte solution with a mediator into the cell.

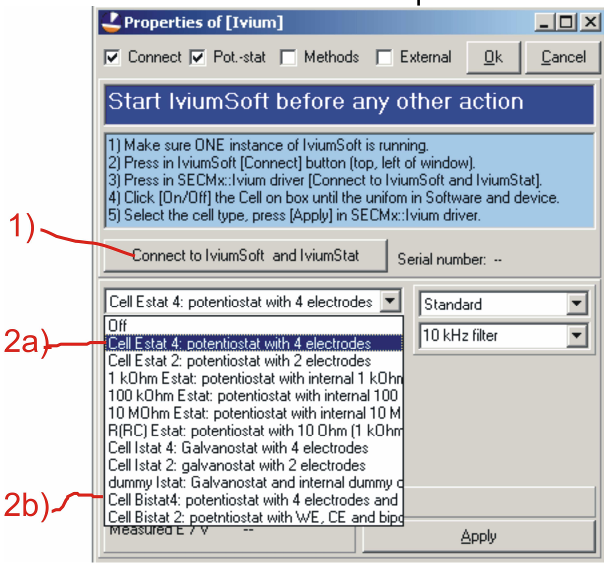
### 1.1.2 Step by Step to an Image

1) Start the IviumSoft software, press ***Connect*** (1), select the high sensitivity option in the IviumSoft (2).




2) Start the SECMx software, select the appropriate user profile.

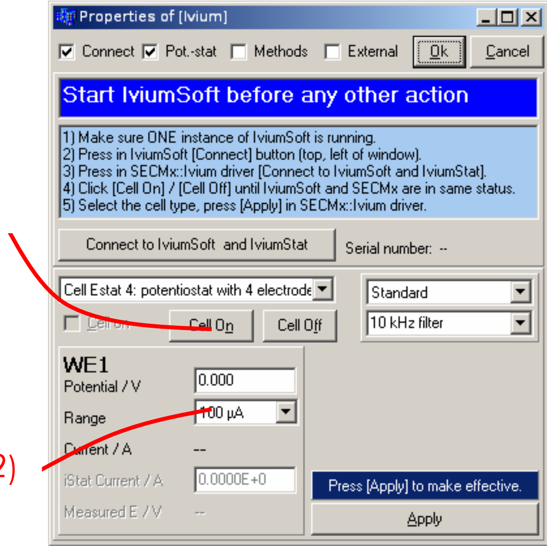
3) If during the loading process the window of the Ivium driver pops up, press ***Connect***. (1). Select the cell type you need. In most cases you will use the monopotentiostat option (2a). If you want to use the bipotentiostat select (2b). The electrode you want to use for potential programs from the IviumSoft should be WE1 (in most cases this will be the microelectrode). For more details see the manual of the Ivium CompactStat.



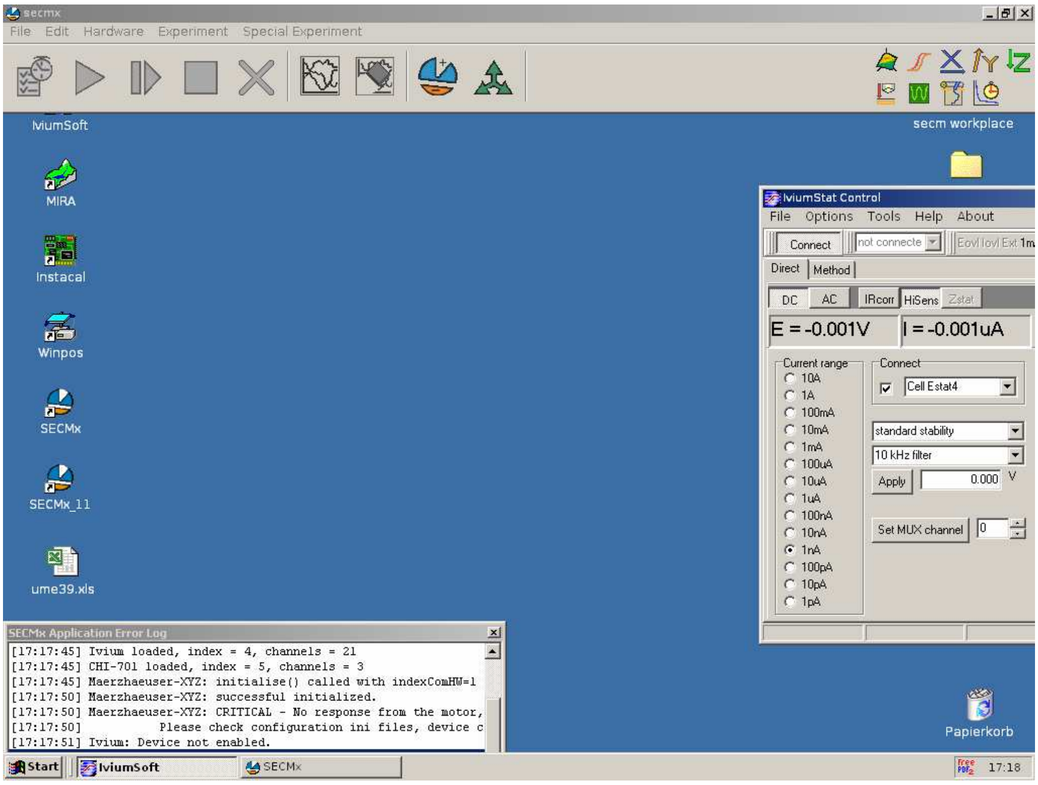

4) Switch on the electrochemical cell (1). Check that the corresponding option control in the IviumSoft will also switch. Then select the current range (2). Select the filter and the potential at which no reaction occurs. Press ***Apply*** and then ***Ok***.

1)

2)




5) Now the loading process of the software is completed and you see the surface. Adjust the screen. It is recommended that you have the control panel of IviumSoft somehow visible on the screen. SECMx main window will occupy the entire upper range of the screen.



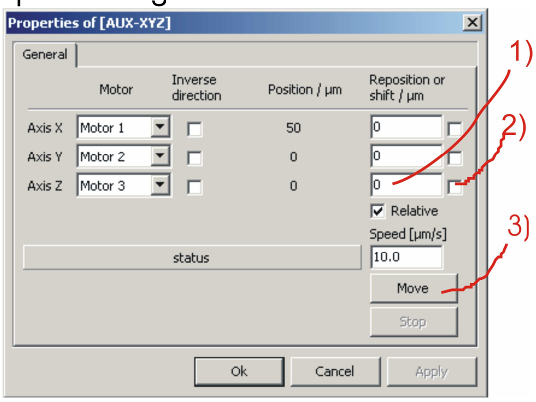

During the first start of the software, you need to tell SECMx at which COM-Port the Märzhäuser positioning system is attached. Select from the menu ***Hardware/Setup Port connectivities***. If you have connected the Corvus controll box to COM1 select in the drop down list for COM1 "Märzhäuser". This information is stored in the devices.ini file. It means that this setting must be made for each devices_xxx.ini file one time.

6) Record a cyclic voltammogram in order to test the connections and the quality of the microelectrode. You should choose in IviumSoft the register ***Method*** and set

the parameters for CV there. You save the result of the measurement as an Ivium data file (*.idf). [You can also run a CV from the SECMx software, but due to the communication overhead, possibilities are more limited]

7) Go back to the SECMx control. Select in the menu ***Hardware/Setup devices***. Select Ivium and press properties. In the setup window of the Ivium device, set the potential of the microelectrode to a potential where a diffusion-controlled reaction occurs at the microelectrode. Press ***Apply***, ***Ok*** and ***Close*** the device selection window.

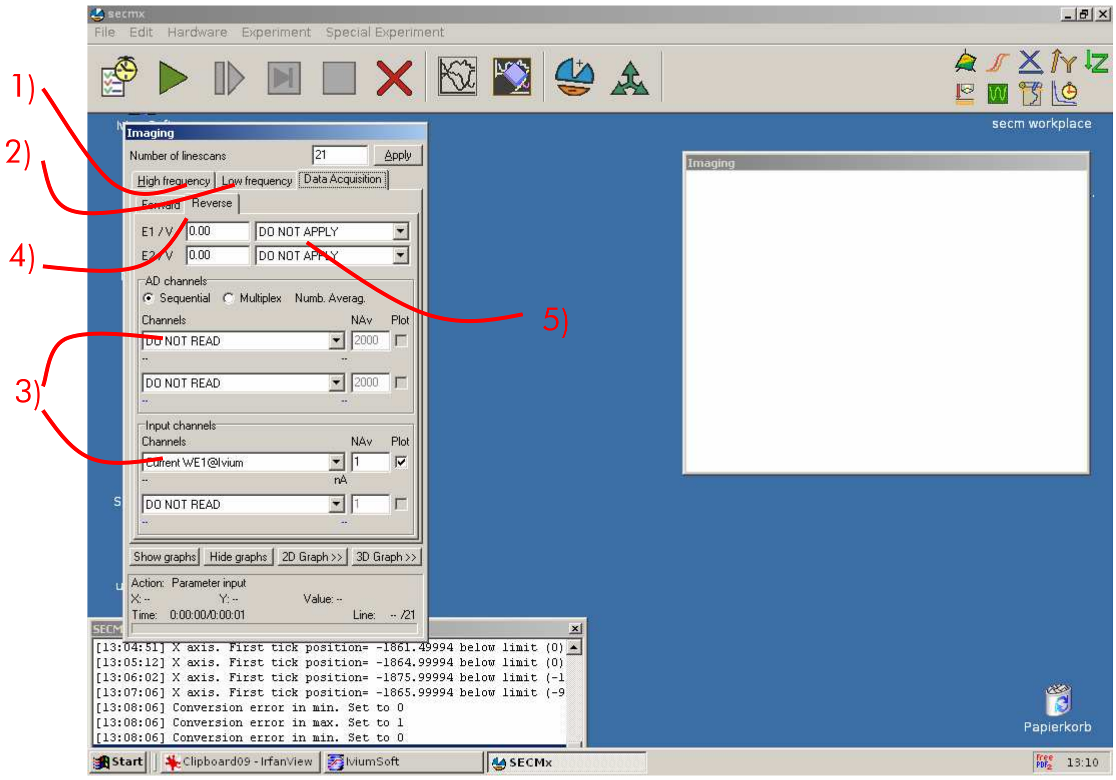
8) Position the tip coarsely with the ***Hardware/Move motors***. Pay attention to absolute and relative positioning.



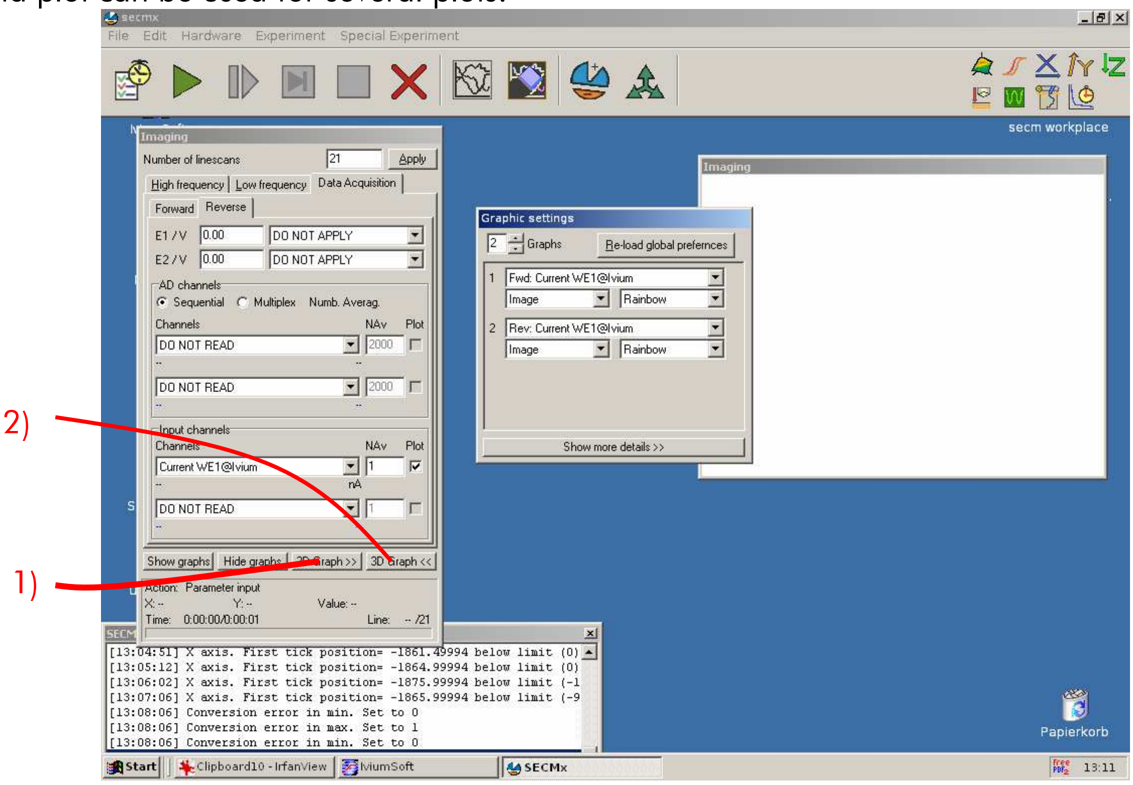

9) Select ***Experiment/Z Line Scan*** and approach the microelectrode to the surface. Interrupt the scan, if the microelectrode touches the surface and retract to the desired distance. Note, that approaching the electrode is the most difficult part of SECM experiments. Depending on the sample, different ways have to be chosen. The detailed explanation of all options is behind the scope of this manual. You must select one motor and at least one data channel. Typically this will be ***Current WE1@Ivium***. The value for **Number of averaging** should be set to ***1*** (when reading IviumSoft!!!!!). If you only read AD channels you may send it to a calibrated value (typically ca. 1724). [Alternatively you may use the routine Fast Approach. It moves the electrode until a preset ratio between the initial current and the present current is reached. If you approach an insulator this ratio could be 0.5, for approaching a conductor it could be 2. The routine is faster than the way described above but requires more understanding of the sample. See Section 1.6]

10) Test with a horizontal line scan ***Experiment/X Line Scan*** or ***Experiment/Y Line Scan*** whether you see the portion of the sample you are interested in.

11) Select imaging. Set the parameters for the forward and back ward scans (1). Set the parameters for the steps in the low frequency axis (2). The channels that can read are controlled by the channel selection drop down (3) boxes separately for forward and reverse scan direction (4). You can apply different potentials during the forward and backward scans via the output option (5). You can also use the output for triggering events like switching on light etc. If you do not want that output of any kind is done during imaging, set the selection box to ***DO NOT APPLY***. In this case all the settings, that were made before the start of the image in the Ivium control window will be maintained during the imaging.



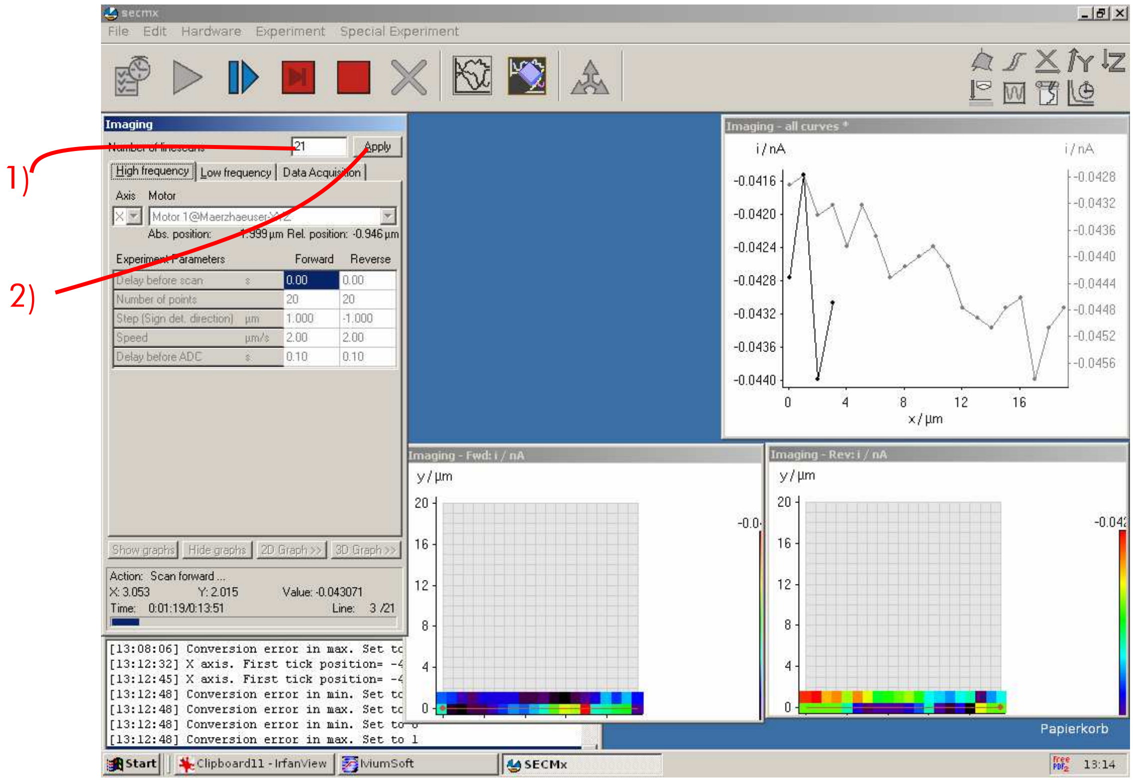

The settings for the graphics are accessible via the buttons **2D Graph>>** (1) and **3D Graph>>** (2). You can select how many 3D plots you want to generate. One data plot can be used for several plots.



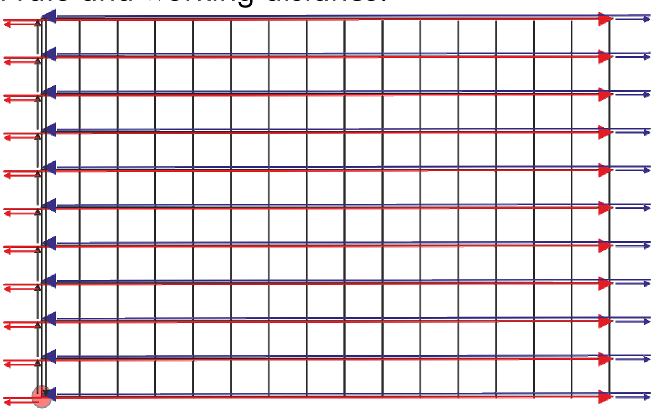

### 1.1.3 Editing parameters during an imaging experiment

Most parameters cannot be edited while an experiment is running. An important exception is the number of line scans during Imaging. This is convenient because only during imaging it becomes obvious that more line scans are needed to capture an entire feature on the sample. When the image is running, the size of the low frequency axis window can be changed (reduced or increased). After editing the number of line scans (1) press ***Apply*** (2).Graphs and time calculation will be updated after the current line scan.

The number of scans can always be increased and it can also be decreased to the number of the currently running line scan.




The movement follows a comb-like movement. The reverse scan retraces the forward scan. In this way two images can be recorded that should be directly comparable. Often there is a small offset between forward and reverse scans depending on imaging mode, scan rate and working distance.

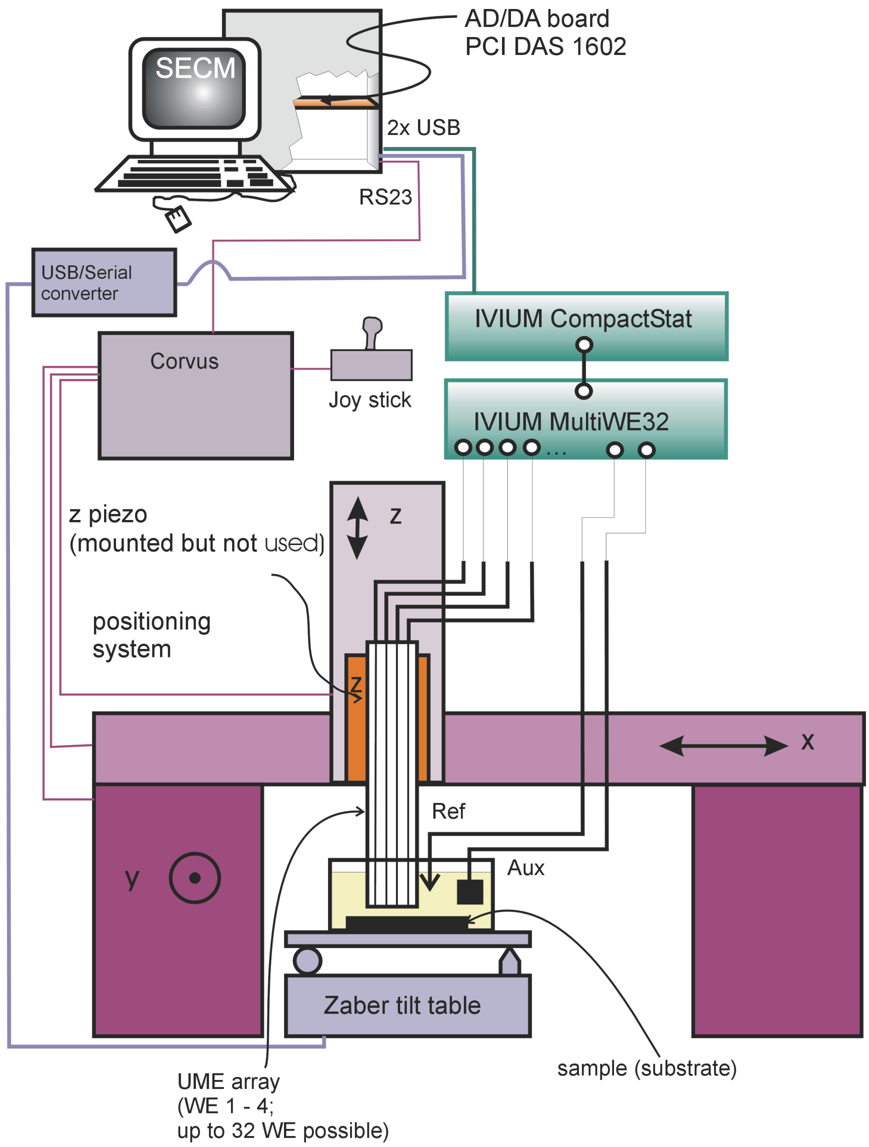


The use of Meander scans is explained in connection with 4D experiments.
The meander scan should not be used for normal imaging experiments, because there is usually a small offset between forward and reverse scans.

## 1.2 Using a bundle of UMEs

### 1.2.1 Setup

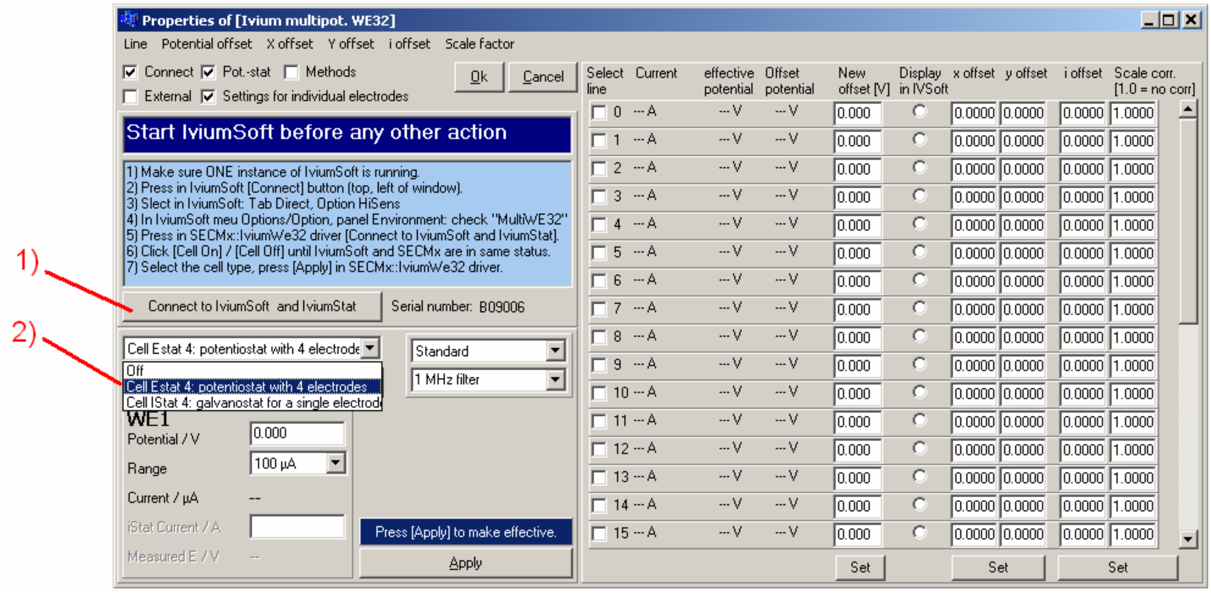
The routines are bdescribed for a setup with a Märzhäuser positioning system and an Ivium CompactStat with an WE32 extension.




### 1.2.2 Step by Step to an Image

1) - 2) See 2.1.2.

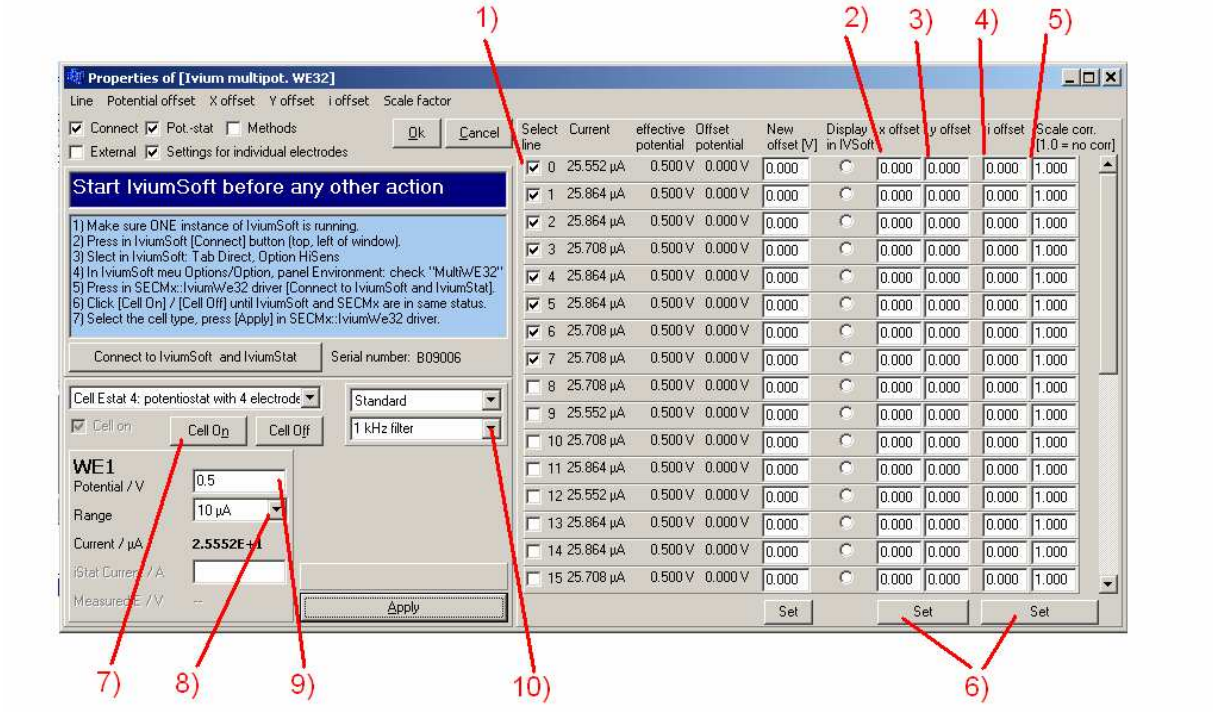
3) If during the loading process the window of the Ivium driver pops up, press ***Connect***. (1). Select the cell type you need (2).




4) If you use an array of microelectrodes you have to check in column **Select line** which Ivium channels/lines you have connected to the channels of the microelectrode array (1). Please note that the channel numbering of SECMx is from **0** to **31** in contrast to Ivium MultiWE32 (from **1** to **32**!). Example: Channel/Line 0 in SECMx means channel 1 in Ivium MultiWE32 and channel/line 23 in SECMx means channel 24 in Ivium MultiWE32. The tips of the array may have positional offsets on the sample and differences in electrochemical response (for instance due to slight variations in their size or working distance):

- *x* position offset (2) (*x offset*, *x offset*[*i*] = 0 means the *i*-th electrode has the same *x* coordinate as electrode[0]),
- *y* position offset (3) (*y offset*, *y offset*[*i*] = 0 means that electrode *i* has the same *y* coordinate as electrode[0]),
- current offset (4) (*i offset*, *i offset*[*i*] = 0 means that no offset is applied to the values coming from sensor *i*),
- correction factor of the sensitivity (scale factor) (5) (*Scale corr.*, *Scale corr.*[*i*] = 1.0 means no correction is applied for electrode *i*).

Only the original measured data are shown in SECMx and are saved in the files! The correction values are written additionally in the saved data files. The saved data files are treated in MIRA afterwards. In MIRA the original measured values are corrected then with the offsets and scale factors. It is easily possible and intended to change the offsets and scale factors in SECMx via ***Hardware/Setup Devices*** and afterwards do fine tuning of the correction values in MIRA. Please read also the MIRA manual. After entering the offsets and scale corrections click the corresponding **Set** buttons (6). Now switch on the electrochemical cell (7). Check that the corresponding option control in IviumSoft and the red LED at the CompactStat will also switch on. Then select the current range (8). Enter the potential (9) and select the filter (10). Press ***Apply*** and then ***Ok***.




5) - 8) See 2.1.2.

9) See 2.1.2. Note that you have to select ***Bundle Experiment/Z Scan Bundle*** and the correct data channel. For bundle experiments with Ivium MultiWE32 two different measuring modes can be used. **Sequential** means that all active channels are measured one after the other each in 20 ms (noise reducing but time consuming). **Multiplex** means that all (always all 32!) channels are measured simultaneously in 100 ms (fast but a bit more noisy), but only the measured data of the channels which are selected in SECMx are stored and saved.

10) Test with a horizontal line scan ***Bundle Experiment/X Scan Bundle*** or ***Bundle Experiment/Y Scan Bundle*** whether you see the portion of the sample you are interested in.

11) See 2.1.2. (2D image bundle). You have to pay attention using arrays of microelectrodes with respect to LF axis! If the LF scan length is larger then the electrode to electrode distance you will scan some areas of the sample double. This means that the first channel of the array will measure areas which the second channel of the electrode has already scanned within its first scans. This doesn't make sense and you should avoid this situation. The following illustration will help you to understand the meaning of **Number of linescans** (*NOFS*) (1) and **Low frequency Step** (*LFS*) (2). The first linescan (HF forward scan) of the image is done at the start position. Before each following linescan the **Low frequency Step** is done. The LF scan length (*LFSL*) is then calculated by *LFSL* = (*NOLS* – 1) * *LFS* and has to be shorter than the electrode to electrode distance (*ETED*, y Offset) of the array:

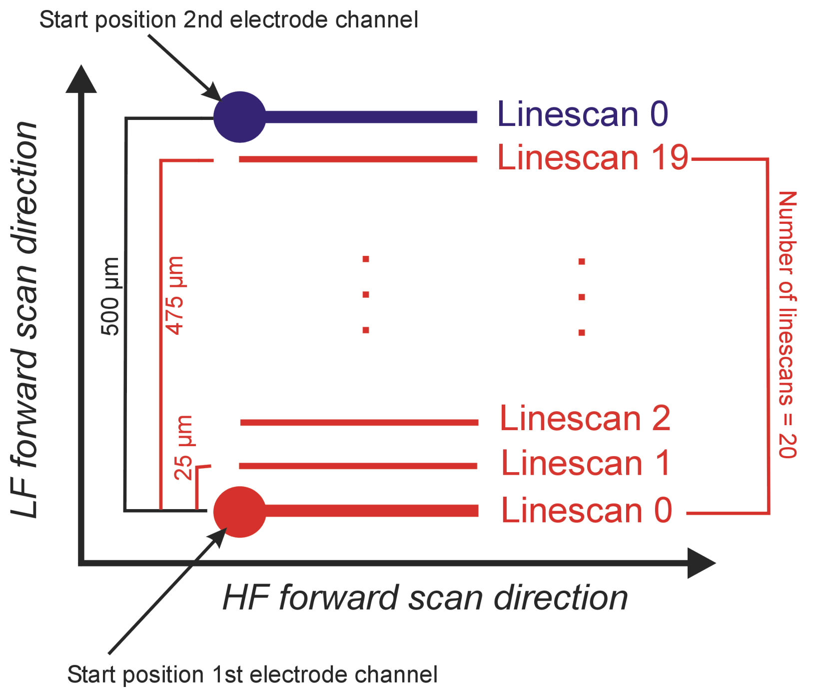
$$(NOLS - 1) * LFS = LFSL < ETED$$

<u>Example 1:</u>

Electrode to electrode distance of the array, *ETED* = 500 µm.
Low Frequency Step, *LFS* = 25 µm.
Number of linescans, *NOLS* = 20.

=> (20 – 1) * 25 µm = 475 µm < 500 µm

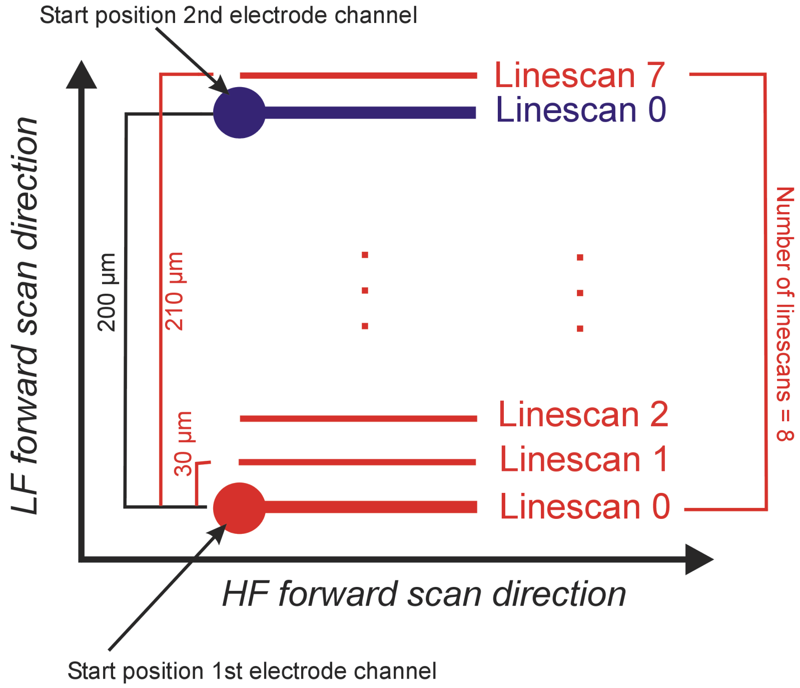
This is ok!




Example 2:
Electrode to electrode distance of the array, *ETED* = 200 µm.
Low Frequency Step, *LFS* = 30 µm.
Number of linescans, *NOLS* = 8.

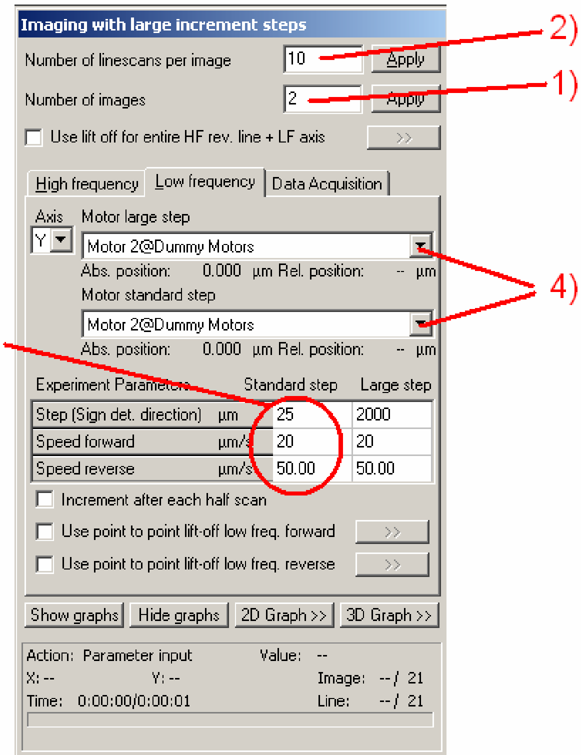
=> (8 – 1) * 30 µm = 210 µm > 200 µm

This doesn't make sense! The last linescan of the first channel scans the area which the second channel already has scanned!



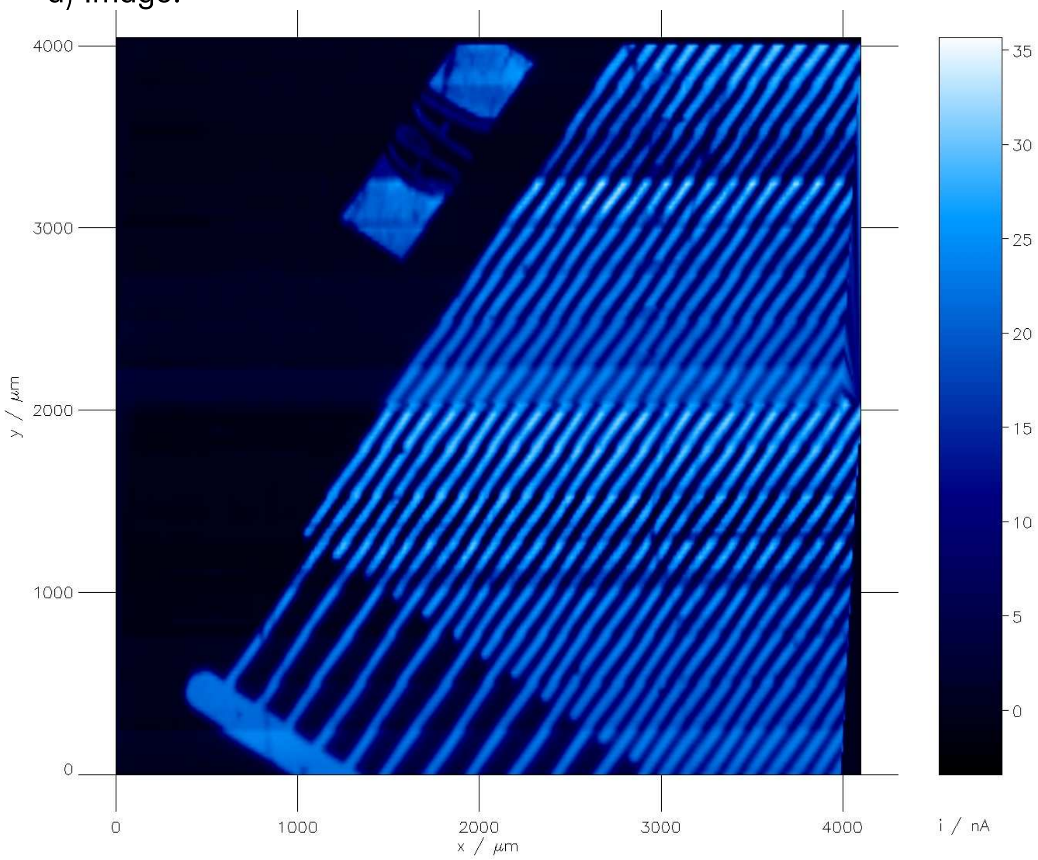

In example 2 you should reduce the number of linescans to 7 because then (7 – 1) * 30 µm = 180 µm is scanned with each electrode which is less than the electrode to electrode distance of 200 µm.

12) If you want to scan areas which are in LF axis larger than the number of array electrodes with electrode to electrode distances provides then you can select ***Bundle Experiment/2D image bundle with large increment step***. You can enter a number of images (1) and a LF large step (2). These images are then made one directly after the other during this experiment. In MIRA these images are merged together to one image. For each single image HF parameters and LF parameters (here Standard step, (3)) are equal to the ones in the 2D image experiment. It is possible to select different motors for standard and large step (explanation will follow in next SECMx manual version). If you use only one motor for LF axis select for standard and large step the same motor (4).




A detailed description of the parameters for standard step and large step are shown in the following figures:

a) Image:




b) Image with explanation:

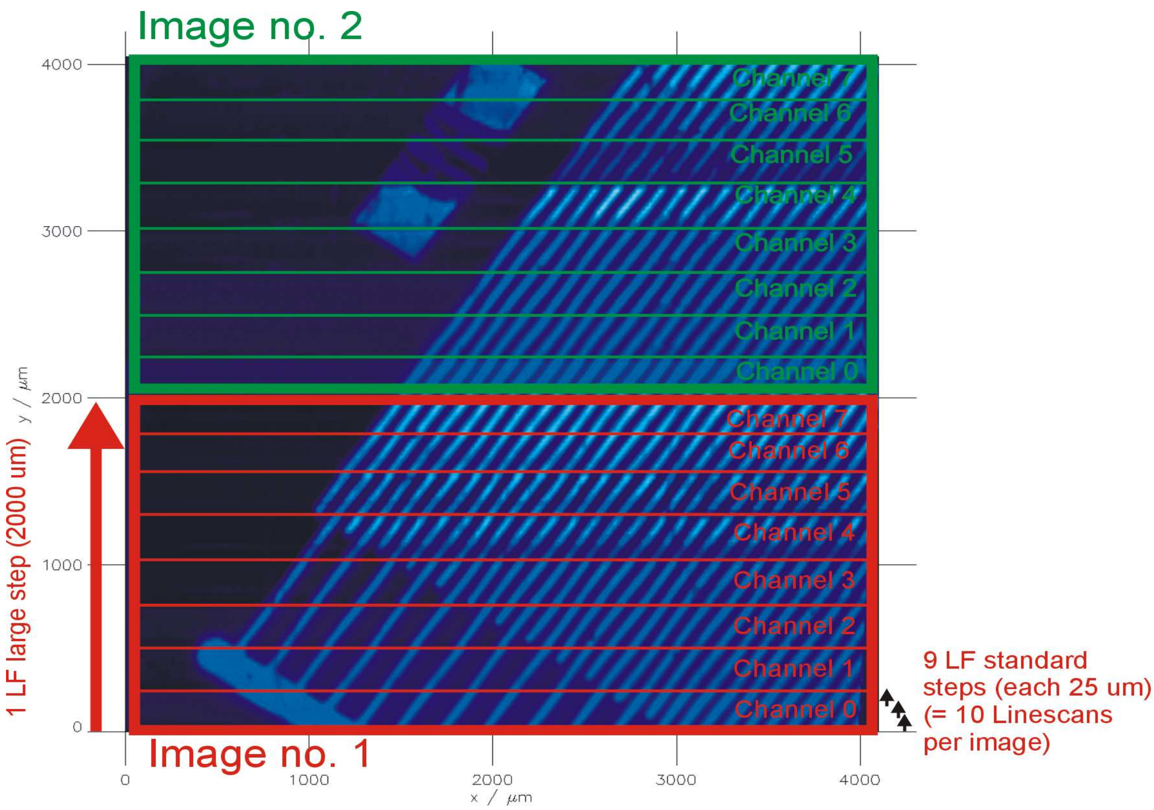
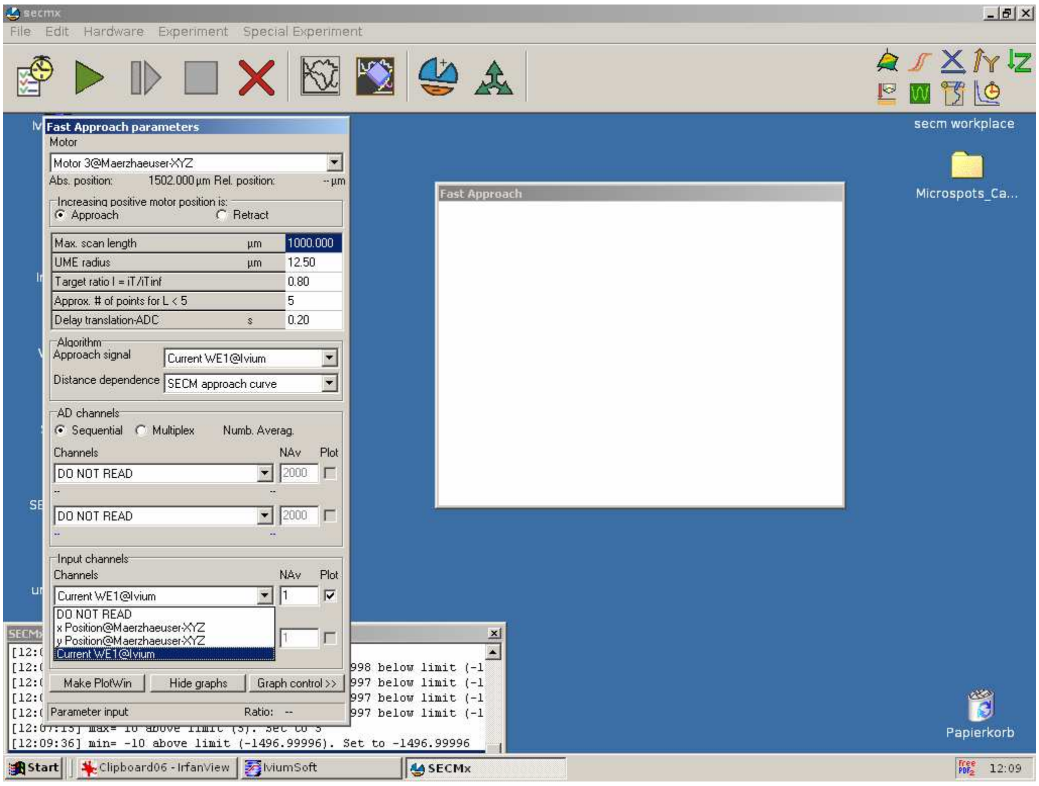


Image no. 2
Channel 7
Channel 6
Channel 5
Channel 4
Channel 3
Channel 2
Channel 1
Channel 0
Channel 7
Channel 6
Channel 5
Channel 4
Channel 3
Channel 2
Channel 1
Channel 0
Image no. 1
1 LF large step (2000 um)
9 LF standard steps (each 25 um) (= 10 Linescans per image)
y / µm
4000
3000
2000
1000
0
0
1000
2000
3000
4000
x / µm

## 1.3 Operation of Fast Approach as an example of a 2D experiment

The Fast approach is explained as an example of 2D experiment. It serves for fast positioning of the probe close to the surface. The movement is slowed down the closer the probe comes to the target position. It is NOT suitable for recording SECM approach curves for kinetic analysis o the sample.




The picture provides an overview for parameters for approaching an impermeable insulator (e.g. a microscope slide). Depending on the mounting of the z motor and increasing z coordinate may mean approach or retract of the probe (here approach).

The next field set the maximum scan length allowed if no surface is detected.

The UME radius is required to predict the next data point and to slow down the movement appropriately close to the target.

The target ratio is the measured current relative to the current in the bulk solution. In order to measure the current in the bulk the probe retracts and then moves forward. The target ratio must be selected according to the sample type (for conductor >1) for insulator < 1). It is recommended to use this routine above sample with either completing inert insulating behavior or with very fast kinetics (bare noble metal).

Approximate number of steps for L < 5, determines how strong the movement is slowed down close top the surface.

Delay before ADC occurs in each method. In order to allow currents through the motors to decay before a current value is measured it is advisable to have a delay. The longer the delay, the longer takes the experiment (particular important in imaging). Therefore, exploring the effect of this parameter is important.

The you select the signal for which the target ratio shall be measured.

Distance dependence currently contains only the SECM approach curves. For the future potentially different distance dependences could be used to predict the probe movement.

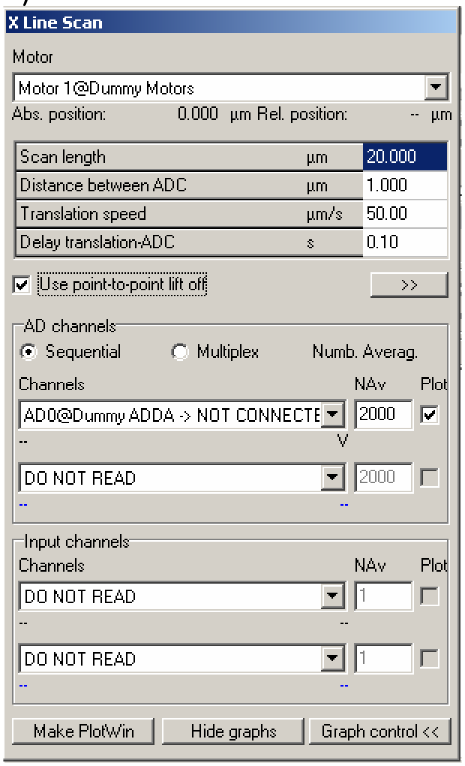
Then you select the AD channels and the In channels to be measured. All measured channels become available for selection as approach channel. You must have selected at least one channel to be measured and you have to have a signal channel for the approach signal.

After start the probe is first retracted to measure the current in the bulk. It then moves forward with steps equal to the electrode radius. If the surface is sensed by a change of the current, the step size is decreased. Usually the probe shoots behind the target and is than slowly retracted and kept on the target value.

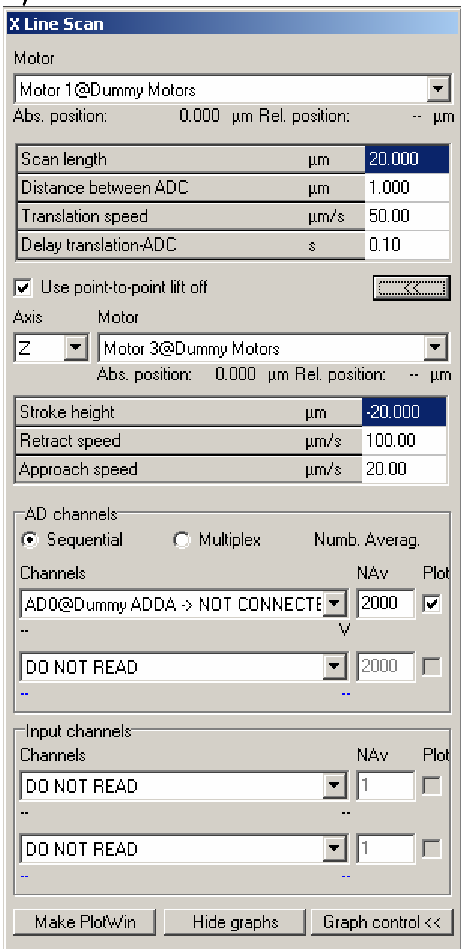
## 1.4 Lift-off mode for line scans and imaging

The lift-off mode provides a way to retract the probe between two measurements points. This can be good for using probes that mechanically touch the sample. In the line scans experiments and images you can check the lift of option and than expand a table to specify the **retract motor** to set the **lift of distance**, the **retract speed** and **approach speed**.

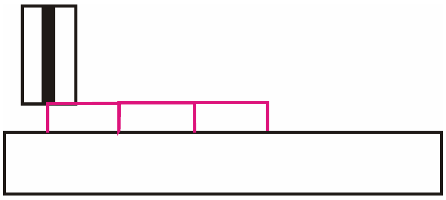
a)




b)

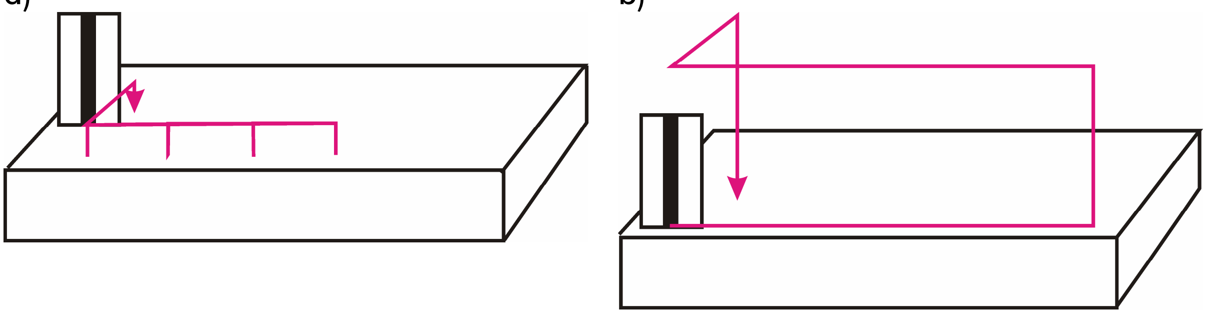



The line lift-off makes a retract and re-approach for each measured point



For images there are two options for the lift off. Technically both can be combined, but at the moment that does not seem to make much sense. The point-to-point lift-off mode (a) approaches the surface between each point. The line lift-off (b) does not change the height during the line scan within one images, but increases the working distances during the backward scan. In both modes the step in the low frequency direction is always done at elevated distance.

a) b)



## 1.5 Some hardware considerations

### 1.5.1 Noise reduction by signal averaging

Many modern potentiostats communicate with the computer via USB, RS232 or other digital data connections. Typically these devices contain an own microprocessor. The Program on the PC loads methods into this microprocessor. The microprocessor carries out the measurement. The PC only handles data storage and interface to the user. The Ivium potentiostat is such a device. In SECMx the read channels of such devices are called In channels, the write channels are called Out channels. (There might be also devices that contain digital and analog channels and may be interfaced in either way.)

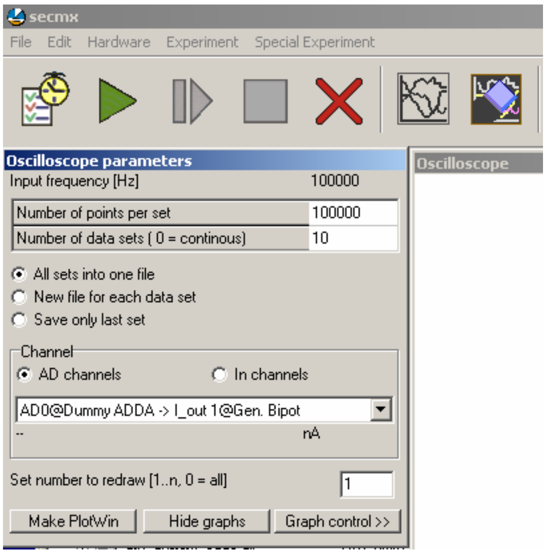
The distinction between the two types of communication is important for a good noise reduction. AD and In channels require a different treatment in this respect. Therefore, you will find separate selection boxes for AD and In channels at all places were signals are to be read. Where data must be written from the PC to external devices you may find alternatively selection tools for DA and Out channels (CV and CA) or all possible write channels are listed in one box (Imaging).

Noise reduction:
The main noise component is typically the line frequency (50 Hz in Europe). One period of this frequency is 20 ms. If the current is integrated over one period of the noise frequency, it almost completely cancelled. For AD channels it means that one has to know the number of AD conversions a AD channel can make at maximum speed within 20 ms. For an AD card with 100 000 kHz frequency, this value would be **NAv** = 2000. Unfortunately, these numbers are not so exact and may depend on the particular PC or version of SECMx. Therefore, it is strongly recommended to calibrate the number (see Section 2.5). The obtained number should be used throughout SECMx for **NAv** of the AD channels. The data should be read by the option **Sequential**, i.e. first all averaging is done from one channel, then from the second and so forth. **Multiplexing** means that reading cycles between all AD channels to read. This reduces the time offset between data points if more channels are read. Because the multiplexer used in the DAS 1602 is much slower than the AD conversion rate, a suitable NAv must be determined separately. It is not even similar to the value for **Sequential**!!!!

Devices with own microprocessors (such as IVIUM) perform these operation independently and typically each data value delivered is already a result of a signal integration. Therefore, further signal averaging does not make sense and the **NAv** parameter should be left at 1.

### 1.5.2 Calibration of NAv (only for systems with AD/DA board)

1) Start the oscilloscope. Collect 10 data sets each with 100.000 data points. Select an AD channel connected to the instrument you are interested in.

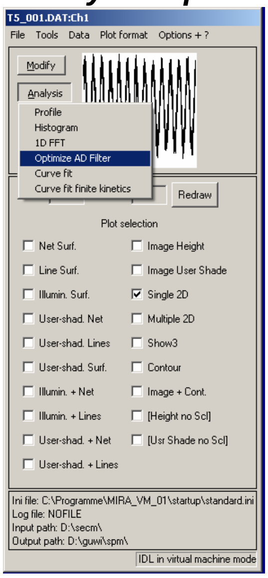



Hint: you should save the data as binary. Otherwise saving may take very long!! Settings can be made under ***Edit/Preferences/Experimental***.

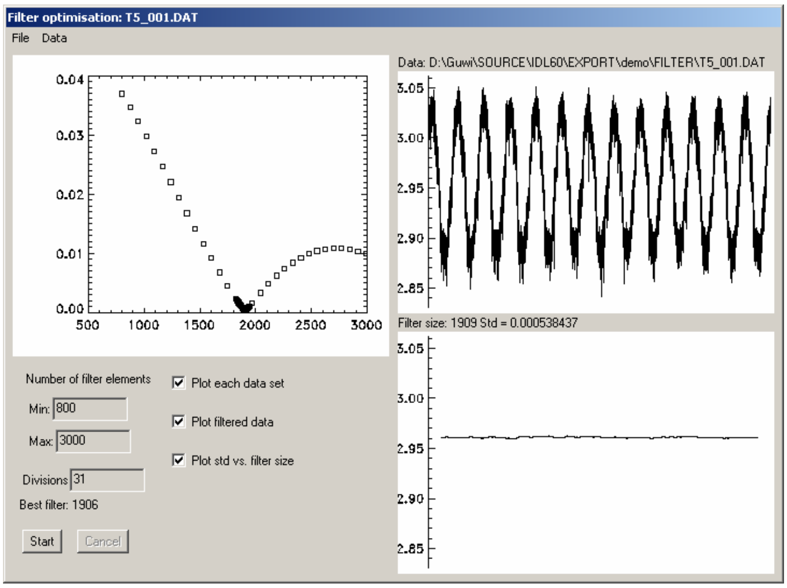
2) Start MIRA.

3) Load the oscilloscope data set into MIRA (Multiplot should be open). Click on one data set.

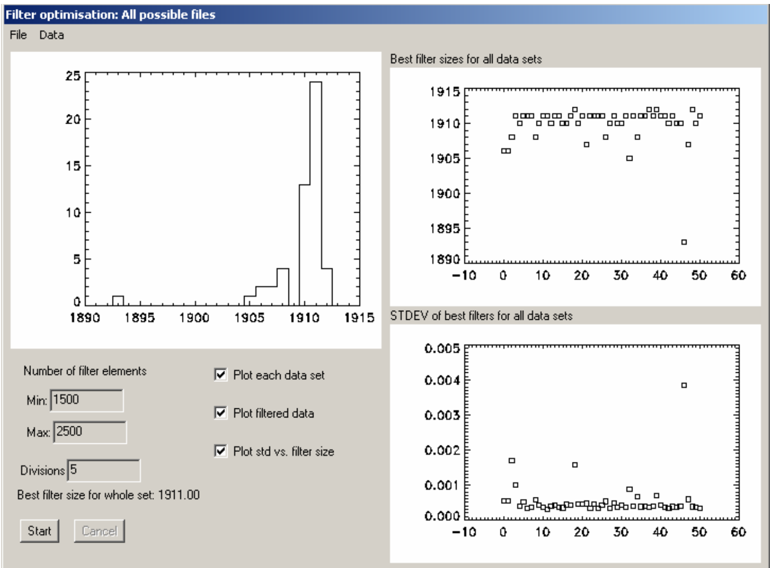
4) Select ***Analysis/Optimize AD filter***.




5) Make the following settings and press start. As the smoothing is calculated, you should see how the smoothing passes through a minimum. The setup is optimized until the best size of the filter is determined. In this particular case it is 1906.




You may want to repeat the procedure with other data sets and make an average of them. This can be done by loading all data sets into the Multiplot window of MIRA. (If you have saved all data sets from step 1 in one binary file, this will automatically be the case.) Then start ***Analysis/Optimize AD filter*** from the Multiplot window. In this case all loaded data sets will be treated and a summary will be generated.

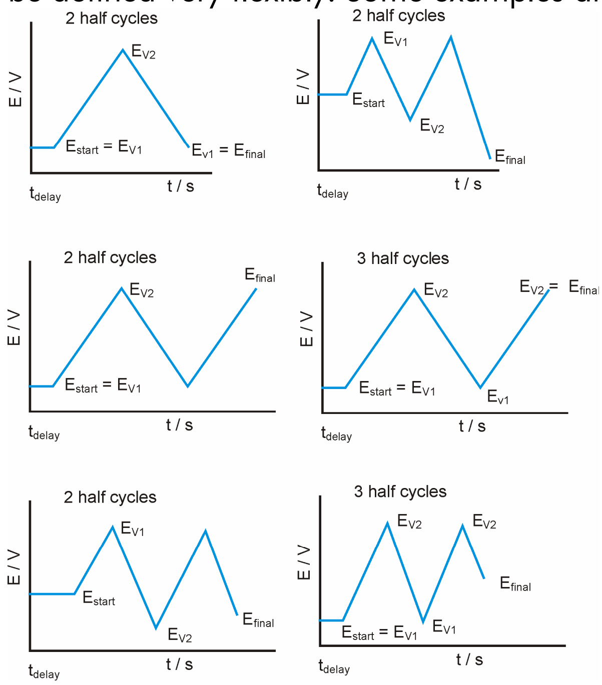


## 1.6 Generic and hardware-specific experiments

### 1.6.1 Generic Experiments

Generic experiments are designed to work with any hardware. Hardware-specific experiments require the availability of particular hardware. For instance, a CV can be performed by incrementing the potential of the working electrode in regular intervals through a command from SECMx. This is a generic way of performing the experiment. However, due to the time delay in passing information through an USB connection, the timing accuracy is limited and rather large potential steps may be required even at moderate scan rates. At analog potentiostat driven by a voltage from an DA board (with much faster communication with the PC), this procedure may yield acceptable results at moderate scan rates. Use of such a routine is fine as long as a steady state response is expected (like an ultramicroelectrode cyclic voltammogram).
Many commercial potentiostats offer the possibility of programming the entire CV before the start. The timing and realisation of the signal is performed by an internal microprocessor of the potentiostats and the resulting data are transferred to the PC on which SECMx is running during or after the potential cycle. That is, SECMx is programming the CV on an microprocessor inside the digital potentiostats and picks up the results if the experiment is done. The definition of the CV depends on how a CV is specified by the commercial potentiostats and therefore varies from instrument to instrument.

Here only the definition of the potential program is given for **generic experiments**.
The CV is defined by the start potential, the first vertex potential, the second vertex potential, the final potential, the number of half cycles, the potential step and the scan rate. HALF cycles refer to complete sweep between the two vertex potentials. Before this, a sweep towards the first vertex potential can be added as well as a segment after reaching the vertex potential for the last time. With those parameters, wave forms can be defined very flexibly. Some examples are give below for typical situations.



### 1.6.2 Special Experiment

Special Experiment routine can be used with a digital (Bi)Potentiostat which can be controlled with its own software, e.g. Ivium CompactStat with IviumSoft. An experiment like cyclic voltammetry can be opened and started via SECMx user interface in IviumSoft. Therefore the experiment is completely controlled by Ivium. This has several advantages, such as the usage of all device specific parameters, e.g. high scan rates up to kV/s, and additional experiments like impedance spectroscopy. All parameters and measured data can be saved in SECMx format. This offers interesting opportunities for Volume scans (see 2.8).

List of integrated Ivium CompactStat experiments (changed 22.10.2010):

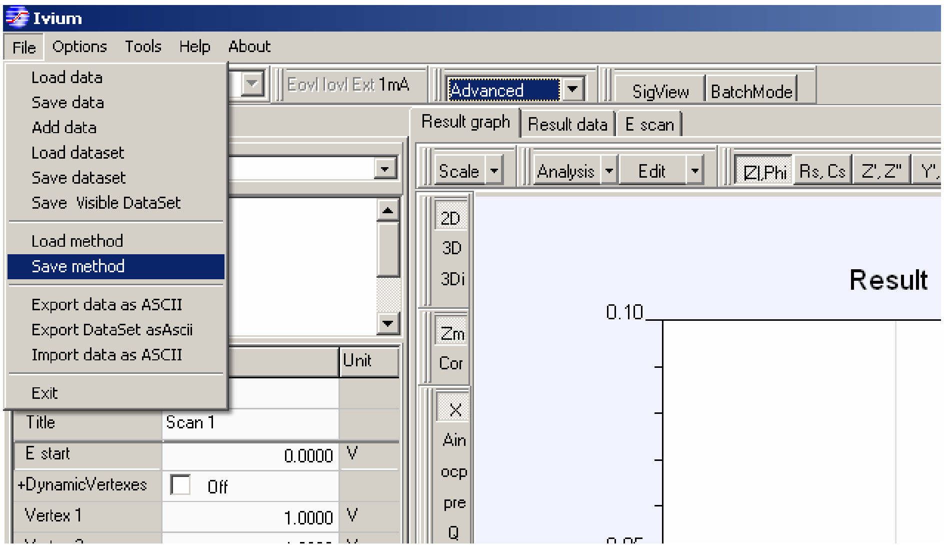
- Transients – ChronoAmperometry
- CyclicVoltammetry – Standard
- Impedance – Constant E
- Impedance – Constant I
- Impedance – PotentialScan
- Impedance – CurrentScan

For the Gamry potentiostats the following special Experiments are available

- Cyclic Voltammetry
- Chronoamperometry

### 1.6.3 Operation with Ivium potentiostats

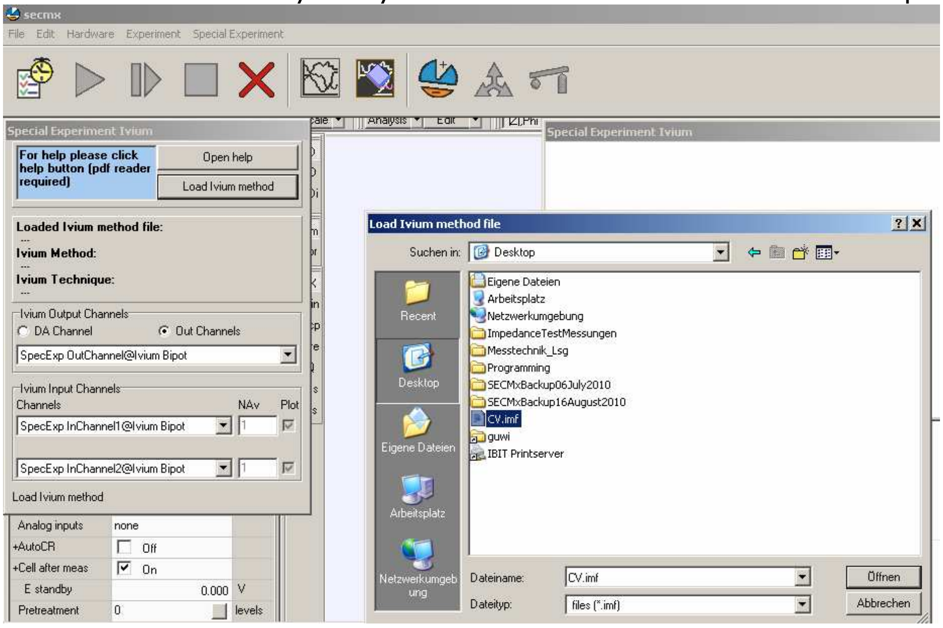
Start IviumSoft before using SECMx! It is strongly recommended to change nothing in Iviumsoft while SECMx is running! Save a method file in IviumSoft in a folder of your choice.




You should save the method file with "cell after meas: ON". Otherwise the cell will be switched off after one experiment and you have to switch it on again in SECMx via ***Hardware/Setup devices*** if you want to make SECMx experiments.

Then start SECMx. Special Experiment will only be available if you have loaded and connected the Ivium Bipotentiostat correctly in SECMx. Select the menu ***Special Experiment/Load Ivium method***. Then load the Ivium method by clicking button

***Load Ivium method*** and selecting the *.imf file. A help pdf file will be opened by clicking the button ***Open help***. The needed Ivium Special Experiment channels are selected automatically. They are different for different kinds of experiments.



Then click ***start***. You can see the plot during the experiment in IviumSoft, not in SECMx. After the experiment has finished you can save it in SECMx and/or in IviumSoft. In the file saved in SECMx all information like the used parameters and so on from the Ivium method file are saved as well as the measured values. Loading the experiment in SECMx is not possible!

### 1.6.4 Operation of Gamry Special experiments

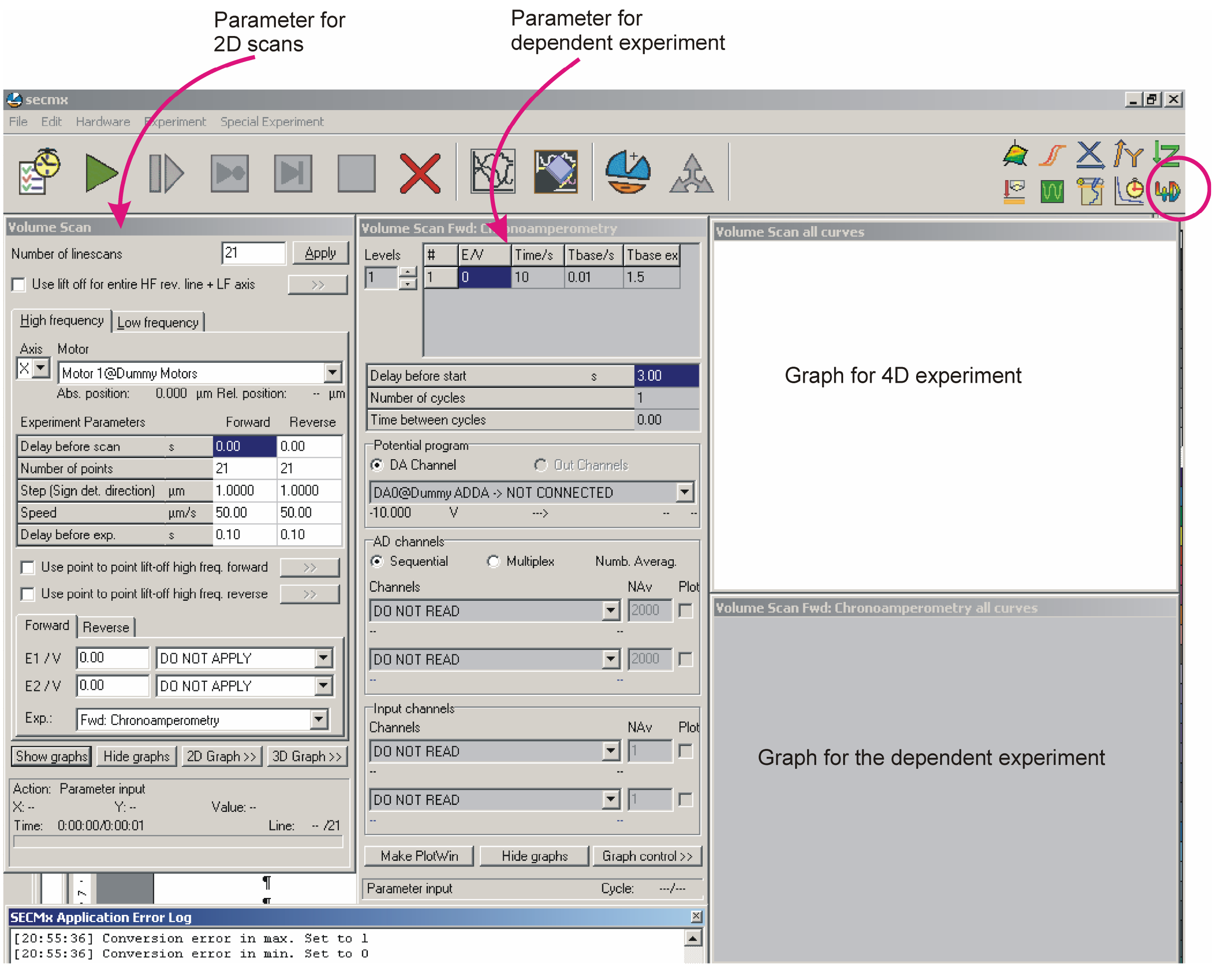
The paramters are set within an Window of SECMx

(Example to be filled)

## 1.7 Four-dimensional measurements (4D, Volume scans)

### 1.7.1 Area scan and dependent experiments

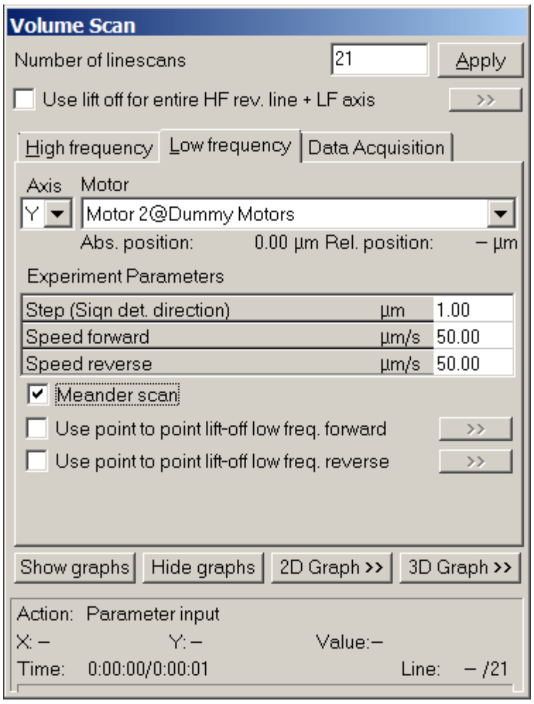
In 4D experiments the probes takes the same way as during in images (including the options of lift-off). At each point of the grid a complete 2D experiment (CV, chronoamperometry, z-linescan) is performed instead of reading a value of the input channels. The parameters of the dependent experiments can be specified in a separate parameters window. The background color of this window is gray in the figure below and can be customized under ***Edit/Preferences***.

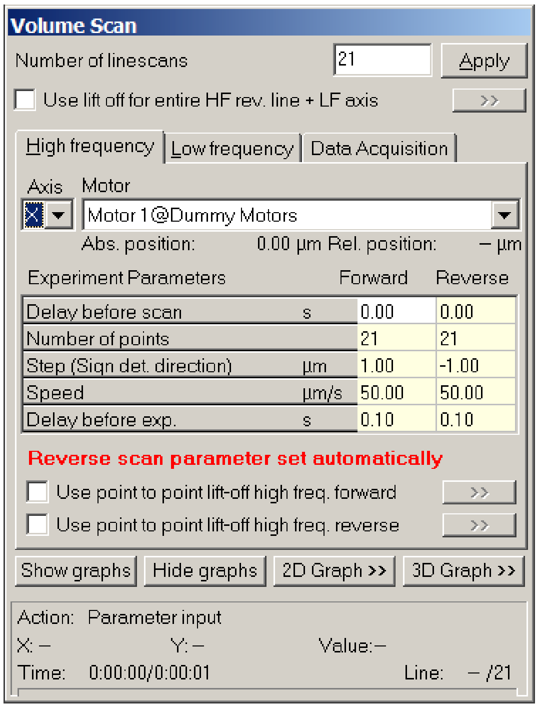



The same color scheme is used to distinguish the graphic windows that refer to the 2D scanning and the individual 2D experiments at each grid point. The image is reconstructed from a specified parameters of the 2D experiment (average, min, max, ...). This mode can be used to perform redox competition mode or surface modifications.

### 1.7.2 Meander scan

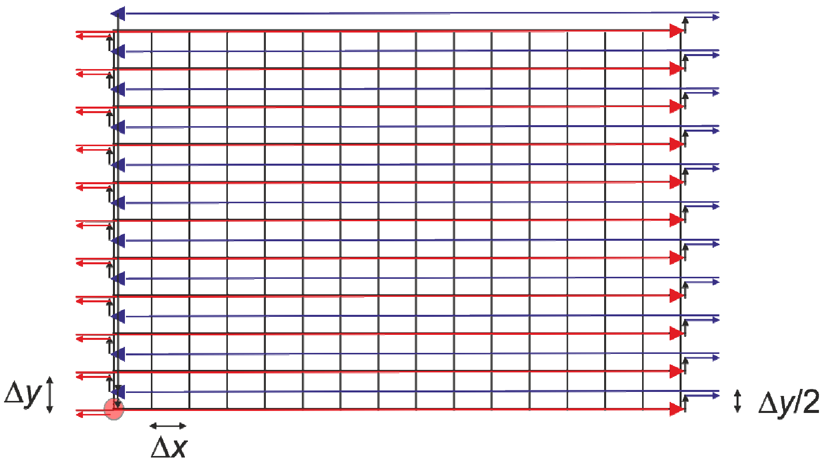
4D experiments can take very long. Therefore, it can be a good compromise to avoiding the retracing and record dependent experiments also in the reverse scans (especially for hopping mode, see next section). The meander scan is caused by setting the check mark on the Tab *Low frequency*. Please not that the background color of the input field on the tab *High frequency* is changing to indicate that the software is in the meander mode.




In the Meander Scan Mode, the data acquisition and scan parameters for forward and reverse scans are identical. Input data for reverse scan are ignored. The parameters of the dependent experiment for the reverse experiment are copied from the dependent experiment of the forward experiment at the moment when the experiment is started. In order to force an intermediate copying of the parameter for the reverse experiment during setup of the experiment use the button for pre-calculate the duration of the experiment in the SECMx menu bar.

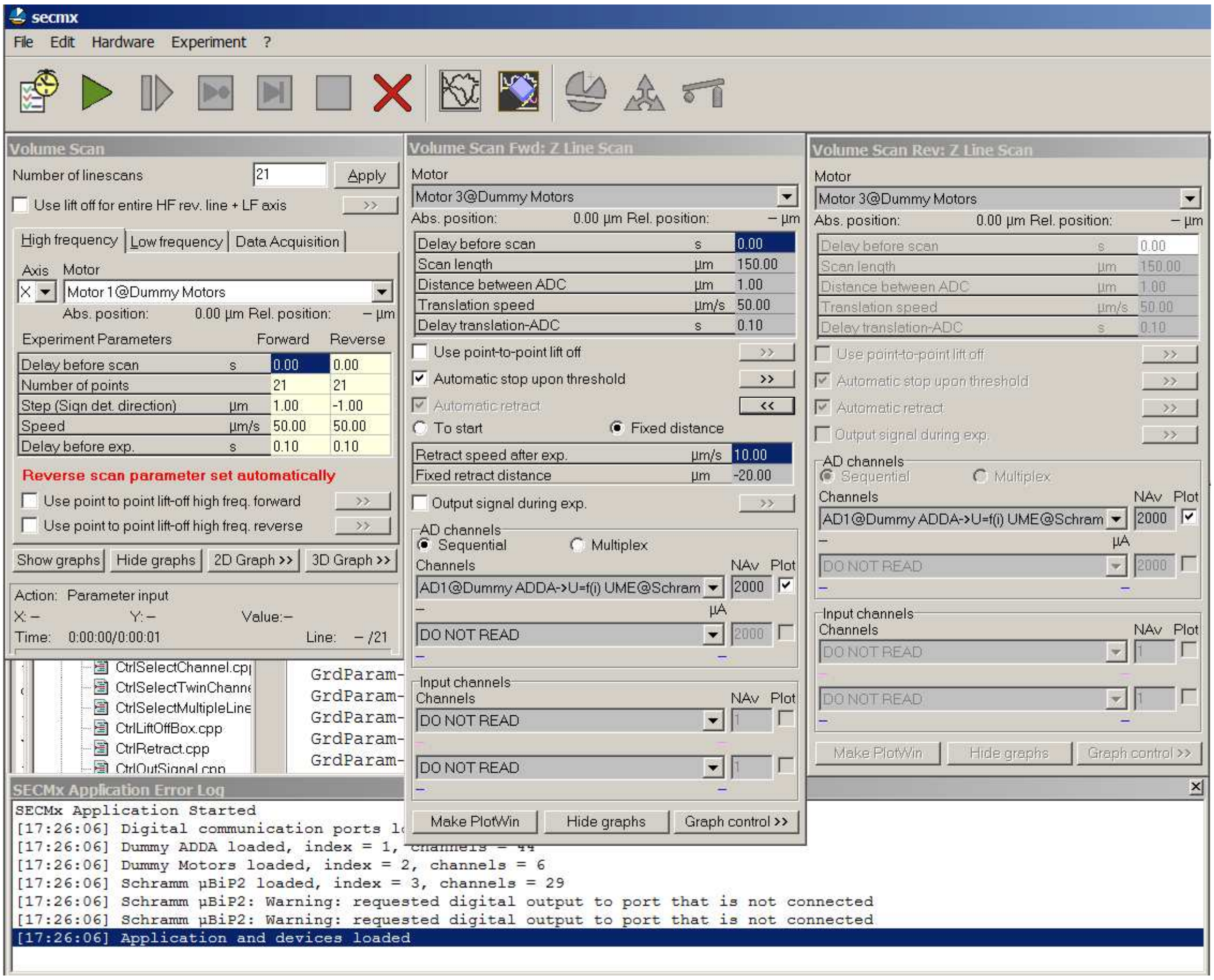
The movement of the electrode is shown below.



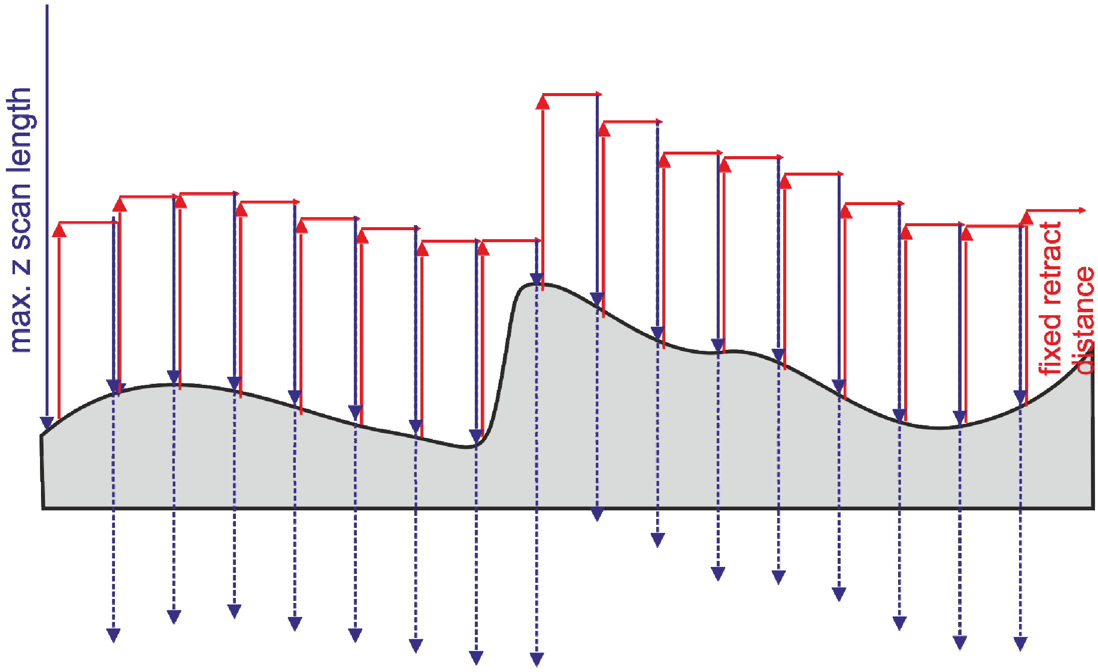

As a result, **two data files** are stored. One datafile contains the data of the forward and reverse scans as separate data blocks. This file can be loaded into SECMx. A second data file with the appendix "_MERGED" contains the data in one data block. It can be read by MIRA for data processing. It cannot be loaded by SECMx.

### 1.7.3 Hopping mode experiments

Hopping mode experiments can be realized when selecting 4D experiments with meander mode and z-scan as the dependent experiment. Within the z-scan parameter the options *Automatic stop upon threshold* detection and *Automatic retract* are selected. Within the expandable Retract control, Retract by *Fixed distance* is selected. The regular scan length is then the maximum scan length and must be larger than the fixed retract distance. In oder to avoid collisions of microelectrode and sample during the reverse scan, there are two possibilities: i) use the line liftoff or ii) use the same dependent experiment (z scan with threshold detection and retraction by a fixed distance for the reverse scan).



Please note that SECMx cannot accurately predict the time for such experiments because the length of the forward experiment cannot be predicted for corrugated samples. The tip movement is given below for one line scan within a 4D experiment.


max. z scan length
fixed retract distance

# 2 Operation with the shear force distance control

The operation of the shear force mode follows closely a paper by Schuhmann et al. which should be cited. We received very detailed information on the Piezo electrode shear force detection. Only the use of the Anfatec controler in connection with the actuators from Physikinstrumente is a new "development" of the Wittstock group. It has the advantage, that the SECM control computer is freed from the task of making the evaluation for the electronic feedback and that the shear-force works completely independent from the SECM main program.

References:


B. Ballesteros Katemann, A. Schulte, W. Schuhmann; Constant-distance mode scanning electrochemical microscopy. Part II: High-resolution SECM imaging employing Pt nanoelectrodes as miniaturized scanning probes. *Electronalysis* 2004, *16*, 60-65.

B. Ballesteros Katemann, A. Schulte, W. Schuhmann; Constant-distance mode scanning electrochemical microscopy (SECM)-part I: Adaptation of a non-optical shear-force-based positioning mode for SECM tips. *Chem. Eur. J.* 2003, *9*, 2025-2033.

U. M. Tefashe, G. Wittstock; Quantitative characterization of shear force regulation for scanning electrochemical microscopy. *C.R. Chimie* 2013, 16, 7-14


For the operation in the shear force mode, connect the instruments in the following way.

a) Perform steps (1) to (8) from section 2. In addition select that ***PI sensor 0*@PI P6xx family** is connected to ***AD0@DAS1602/16*** (in ***Hardware/Setup AD/DA connections***). (the number of the PI ensor may vary depending on the setup)

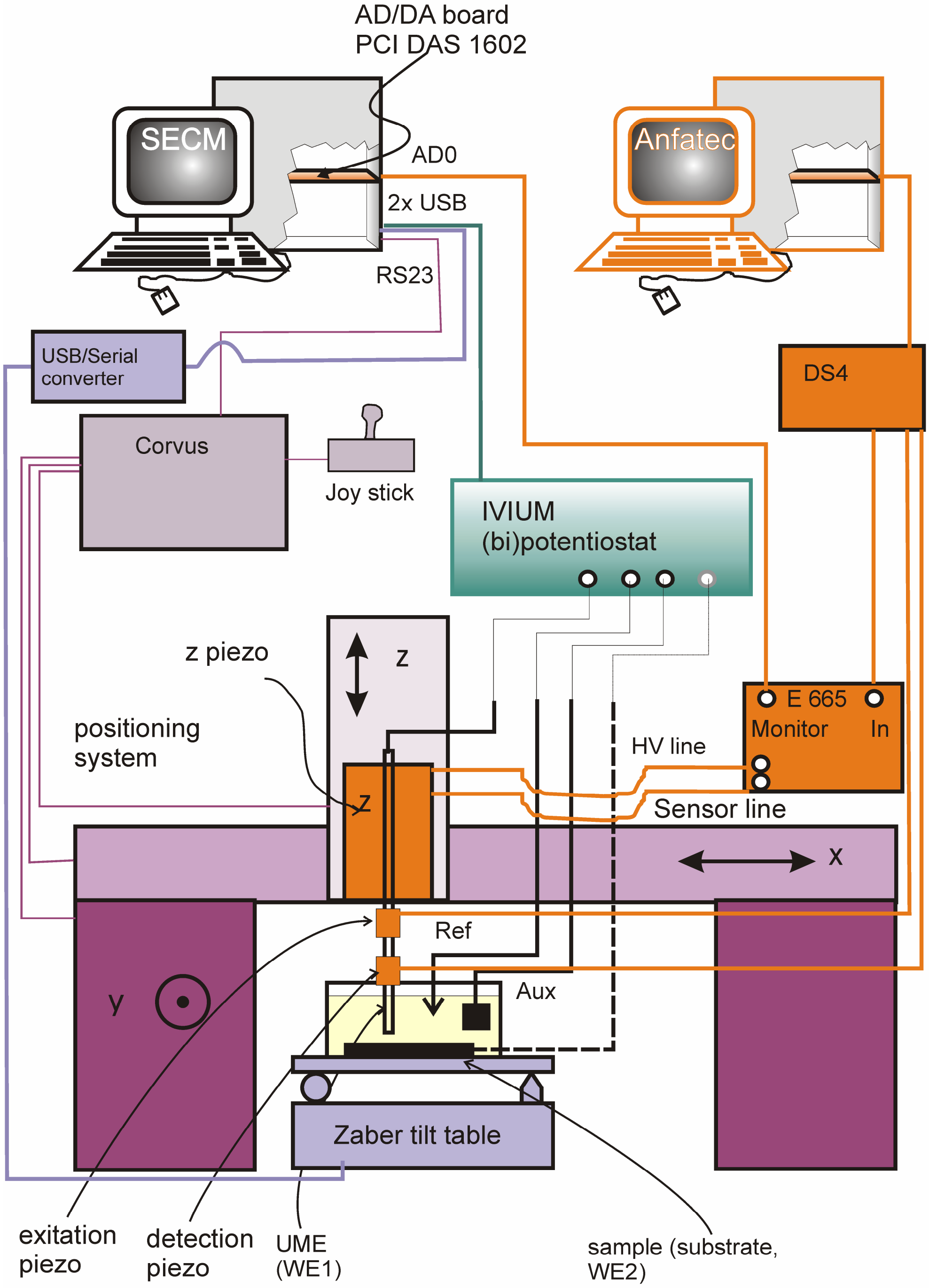
b) Record a vibration spectrum of the pulled microelectrode about 1 mm above the sample.

c) Record an approach curve (Experiment/Z Line Scan) and read the current Current WE1 (not connected): Ivium

d) Record another vibration spectrum with the tip within 1 micrometer off the surface. Overlay the two vibration spectra and select the excitation frequency at which the highest difference in amplitude or phase is observed.

e) Retract the microelectrode about 100 $\mu$m from the surface.

f) Give a setpoint. The setpoint is the ratio of the amplitude far away from the surface and close to the surface. The change in the signal may be very small (only 2 % giving a setpoint of 98 % for instance). After setting the setpoint, the piezo should expand towards the surface up to the maximum position. Since the sample is not reached it will stay at far most extension.




g) Now perform a ***Experiment/Z Line Scan***. Once the microelectrode comes close to the surface, you should see how the piezo retracts while the motor of the Märzhäuser system drives the mircoelectrode towards the surface. This is displayed on the screen of the Anfatec PID controller. If the piezo position is about in the middle of the piezo movement range, stop the movement of the Märzhäuser z motor. Now perform horizontal line scans with the spacing and speed you would use for imaging and optimize speed and PID settings. This requires experience and no clear rule can be given.

h) You can perform imaging and read ***Current WE1@Ivium*** (= reactivity) and ***AD0@DAS1602/16*** (= topography).

# 3 Procedure Checklist

In order to operate the SECM efficiently, you should know what the following terms mean within the SECMx program

- ☐ What is a 'channel'
- ☐ What is a 'device'
- ☐ What is a 'bundle'
- ☐ What is a 'mixed bundle'
- ☐ Which channels or devices require signal averaging? Which parameter controls signal averaging? How is signal averaging optimized?
- ☐ What are 'generic experiments'?
- ☐ What are 'special experiments'? What are their advantages?
- ☐ What is a generic cyclic voltammogram. How do I define the potential program?
- ☐ What is a line scan?
- ☐ What is the difference between a 'z-scan' and a 'fast approach"? Which of them should I use when I want to compare the approach curve to the theory for steady state response?
- ☐ How are the parameter defined for Imaging experiment?
- ☐ What is the Meander mode?
- ☐ What is a 'Volume scan'?