

1 Introduction

1.1 Field Effect Transistor and Bipotentiostats

Field Effect Transistors (FET) are essential components in modern electronics. Furthermore, they have been used for chemical sensors. Sensors based on FETs stay an emerging field.

FETs have variable resistance depending on an electric field applied to them. Usually, a voltage is applied between the two terminals on the opposing ends of the material with the variable resistance, typically a semiconductor (see Figure 1). The two terminals are called source and drain. A voltage is applied between them, the source-drain voltage V_{SD} .

Another voltage is applied perpendicular to the material with variable resistance. This is the gate voltage V_G or depending on between which points it is applied V_{SG} or V_{DG} . The gate itself is not connected to variable resistance element, so only the electric field of the gate influences the FET.

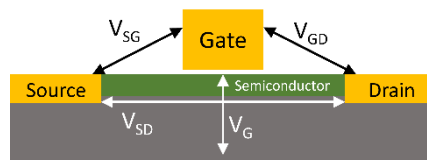


Figure 1 schematic representation of a FET and the applied voltages

A change in V_G leads to a change in resistance between the source and the drain. This means with a constant V_{SD} , but a varying V_G the current between the source and drain I_{SD} will change. Depending on the FET's parameters this process can be used for signal amplification.

Due to their use in chemical sensors, FETs are interesting for electrochemists as well. When starting to investigate FETs, researchers consider the purchase of equipment to characterize their FET-based sensors an obstacle. Most likely every electrochemical lab is already equipped with the necessary tools.

To characterize a FET, you require control of two potentials, V_{SD} and V_G , plus you need to measure one current, I_{SD} . The gate current I_G is ideally 0, but in real setups there is a leakage current, usually negligible. Recording I_G provides information about the FET's condition and thus advised. This can be performed with a bipotentiostat, i.e. a potentiostat with 2 WEs, 1 RE, and 1 CE.

1.2 FET-based sensors

FETs can be used for amplification by using the voltage to be amplified as V_G and controlling the amplification by applying a high V_{SD} . Commonly used for this amplification in electronics are metal oxide semiconductor FETs (MOSFET).

This amplification is not limited to pure electronics. The origin of the gate voltage could be an external potentiometric probe, for example, a glass membrane electrode used for pH measurements. If a probe is connected to the gate connection, an Extended Gate FET (EGFET) is created. This creates a situation where the pH electrodes potential is added to V_G and thus making V_G pH dependent.

For miniaturization, it is handy to get rid of the probe. For that purpose, the gate's surface is in contact with the sample solution. The V_G is applied between the gate material and a reference electrode in the sample solution. The gate's surface can be modified to interact with a specific analyte. This interaction should change the potential across the interface. To stick with the example of a pH electrode, the gate's surface could be covered in a thin silicon oxide layer acting like a glass membrane. The potential across the glass membrane is added to the applied V_G and thus the I_{SD} becomes pH dependent. Such a sensor is an ion-selective FET (ISFET). These systems can also be based on biomolecules like antibodies, aptamers, enzymes, DNA, etc. These sensors are declared analog to a biosensor as bioFET. If the surface is modified with non-biological molecules the device is a chemFET.

FET research is looking into semiconducting materials that are already sensitive to specific analytes, which leads for example to graphene-based FETs (GFET).

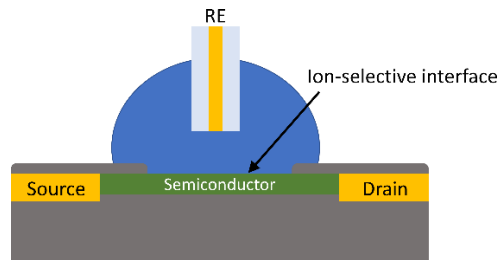


Figure 2 schematic representation of an ISFET

A more detailed description of each FET type is included in an article recently submitted for peer review in an open-source journal:

<https://chemrxiv.org/engage/chemrxiv/article-details/6284b89f6b12b6bc00760e65>

(DOI 10.26434/chemrxiv-2022-lv0k6)

This article is the result of a cooperation between Filipiak et al. (IPC PAS, Warsaw, Poland) and PalmSens.

1.3 FET Characterization

There are two important graphs in FET characterization: the output curve and the transfer curve.

The transfer characteristics of a FET are measured by applying a constant drain-source voltage (V_{SD}) and measuring the drain-source current (I_{SD}) while sweeping the gate voltage (V_G). The output characteristics are evaluated by applying a constant V_G bias and measuring I_{SD} changes while sweeping V_{SD} .

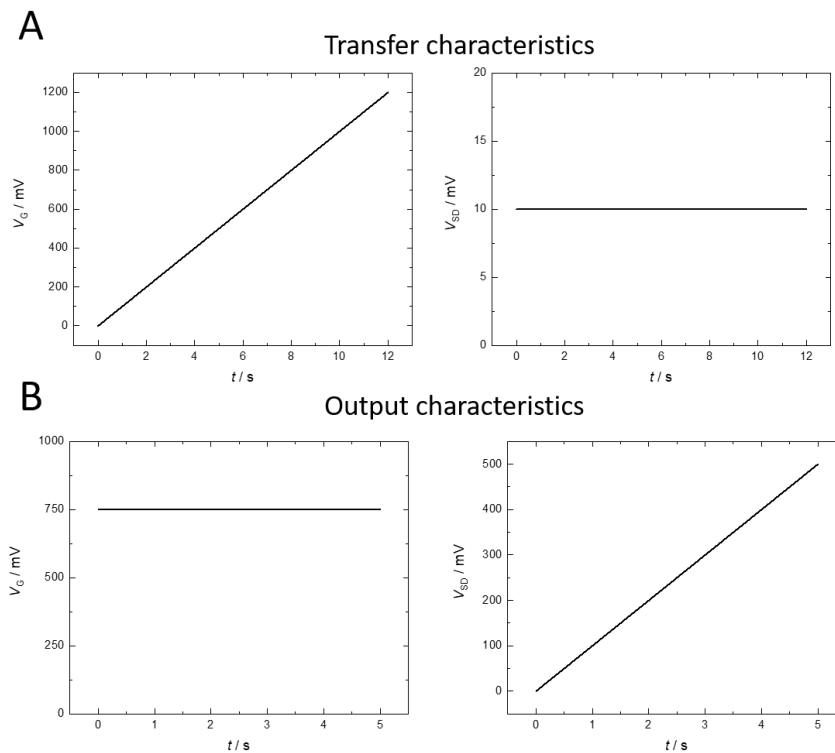


Figure 3 potential profiles of V_G and V_{SD} during the recording of a transfer curve (A) and output curve (B) source: DOI 10.26434/chemrxiv-2022-lv0k6

A more detailed description and examples are included in an article recently submitted for peer review in an open-source journal:

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2 EmStat Pico

As described in chapter 1.1 for FET characterization two different potentials are controlled, V_{SD} and V_G , the source-drain current I_{SD} is recorded, and optionally the gate current I_G is recorded.

A single-channel potentiostat won't be able to perform these measurements. A bipotentiostat or two single-channel potentiostats are required.

An EmStat Pico has 2 WEs, 2 RE, and 2 CE connections because it has 2 channels. When switched to the bipot mode it has effectively 2 WEs, 1 RE, and 1 CE. The first WE can be used to control a variable potential and the second WE for applying a constant potential, while both electrodes record at the same time currents.

Using a single device with perfect synchronized potential control and current recording for the measurement is practical and cost-effective. An advantage of the EmStat Pico is the [MethodSCRIPT](#) capability. It allows effectively to have auto-ranging on both working electrodes. Standard bipotentiostat measurements allow the current range at the first WE to be optimized (auto-ranging), but the current range at the second WE is fixed. The MethodSCRIPT code for this application note (see Annex 4.1) includes a loop that optimizes the current range for the second WE, too.

2.1 Setting up the Connections

The second WE can either apply a constant potential or follow the change at the first WE. For the transfer and output characterization, two different potentials need to be scanned, V_G for the first and V_{SD} for the latter while the other potential stays constant. A combination of a potential sweep and constant potential can only be performed with the first WE sweeping and the second WE at a constant potential. Thus, for measuring the transfer curve and the output curve, the connections need to be rearranged when the characterization method is changed.

For measuring the transfer characteristics connect the WE0 to the gate, the WE1 to the drain, and combine the RE0 and CE0 at the source (see Figure 4). WE0 will be assigned as the main channel and WE1 as the bipot in the MethodSCRIPT.

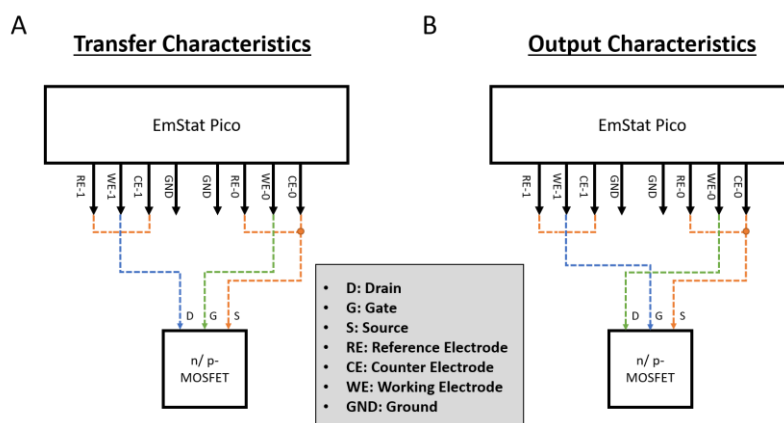


Figure 4 Connections of the EmStat Pico for a transfer curve (left) and output curve (right) source: DOI 10.26434/chemrxiv-2022-lv0k6

For the output characteristics swap the WE0 and WE1, i.e. connect the WE0 to the drain, the WE1 to the gate, and combine the RE0 and CE0 at the source (see Figure 4)

To perform your measurement, you need to execute the MethodSCRIPT code that you can download as [a txt file](#) and [method file](#), or copy-paste from the annex of this application note (see page 8).

When you set up the EmStat Pico take into consideration that programmers start to count with 0, thus the first WE is on channel 0 and the second electrode is on channel 1. You can swap the two channels if you want when setting the modes in MethodSCRIPT.

2.2 How to add a MethodSCRIPT in PSTRace

The MethodSCRIPT code presented in this application note allows the EmStat Pico to perform auto-ranging on both channels, which means that the EmStat Pico is capable of measuring currents accurately over several magnitudes. Depending on the connections, the script allows recording transfer or output curves (see Figure 4).

To add a MethodSCRIPT code to PSTRace's sandbox, you need to open PSTRace and connect an EmStat Pico, Sensit Smart, Sensit BT, or the virtual EmStat Pico. Or you go to *Tools – General Settings...* in the top menu and check the box *Show MethodSCRIPT Editor*.

The button *Show MethodSCRIPT* will appear. Click on that button and the MethodSCRIPT corresponding to the parameters in the Method Editor will appear (see Figure 5).

Click on the *Open in Editor* button and the editor will open. Here you can change the MethodSCRIPT code. If you copied the FET MethodSCRIPT code from the Annex (see page 8) or txt file to your clipboard, you can just click into the editor field, press Ctrl+A, then Ctrl+V, and the MethodSCRIPT code should be copied to your sandbox (see Figure 6). Don't forget to make sure there are 2 empty lines at the end of the script. If you downloaded the method file, just go to *Method – Load* and select the method file. To adjust the MethodSCRIPT code, click on *Open MethodSCRIPT Editor*.

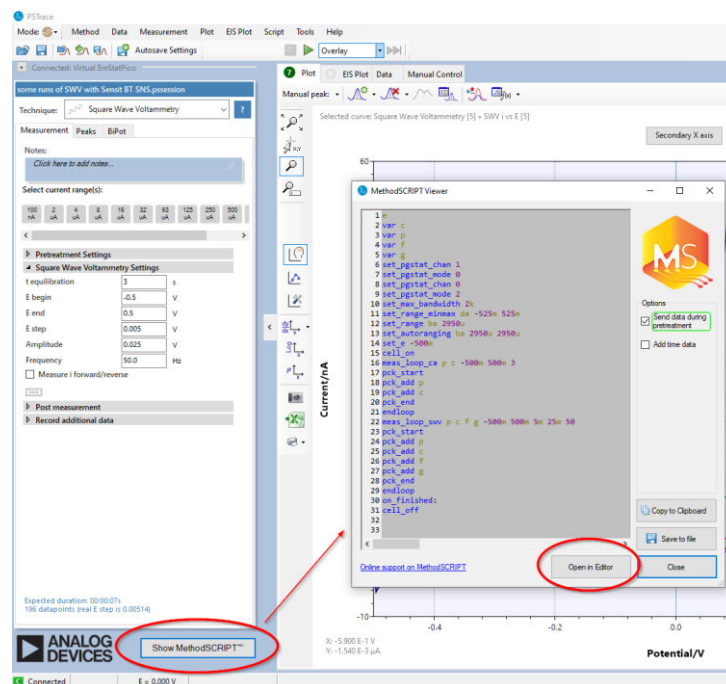


Figure 5 How to Open the MethodSCRIPT viewer and change to MethodSCRIPT Editor

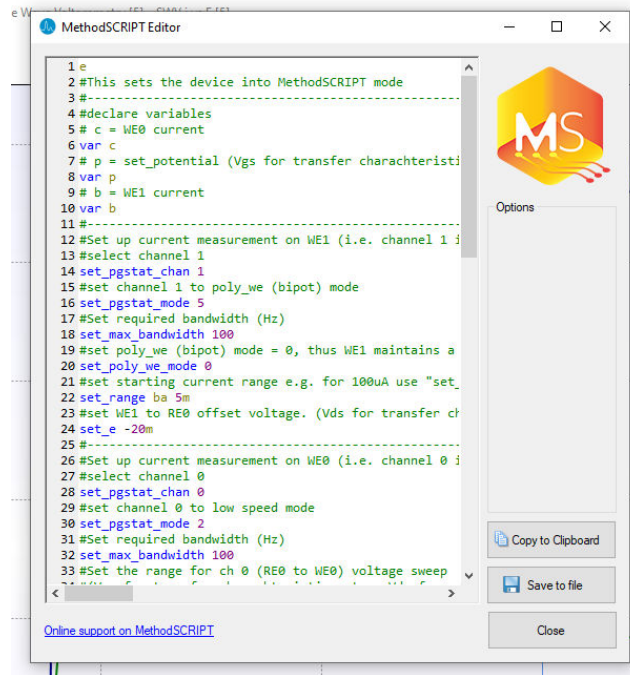


Figure 6 MethodSCRIPT Editor

2.3 How to set the Parameters in the MethodSCRIPT

The MethodSCRIPT is a script and thus does not offer a convenient mask like the usual PStace interface. To compensate for this inconvenience this MethodSCRIPT code example has a lot of comments, which should make it easy to adjust the parameters as required. Here some of these parameters will be pointed out.

In this script the WE0 is used as the electrode with the potential sweep, so the connection schematics in Figure 4 apply. One obstacle is that programmers start to count with 0. So, the first channel is 0 and the second channel is 1. This can easily lead to mix-ups when WE1 is mentioned. Is it WE1 of WE0 and WE1, or is it WE1 of WE1 and WE2?

First, the constant potential can be set. To describe the script the line numbers as shown in the MethodSCRIPT Editor are used. In line 24 you set, as described in line 23, the constant potential for the bipot electrode (WE1) (see Figure 7). Usually, the value is the potential difference between the reference electrode (RE0) and the bipot working electrode (WE1) in mV, for example, if you want to start 1.5 V below the RE, i.e. -1.5 V, you would enter *-1500m*.

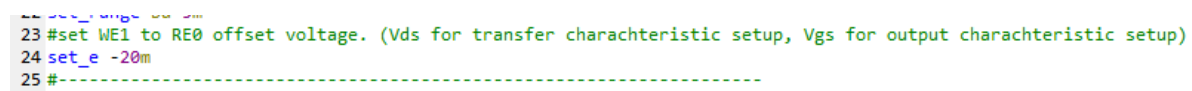


Figure 7 lines 23 to 25 of the MethodSCRIPT code, setting the constant potential of WE1

Lines 41 to 54 are all required to add an equilibration time, just like the *t_{equilibrium}* in PStace, and the comments in these lines explain exactly that. The script starts with both channels in the 5 mA current range. These lines apply a constant potential and adjust the current range to match the measured current for both working electrodes.

In line 46 you set the applied potential for the WE0, the duration of the applied potential, and how often it is recorded (see Figure 8). If your FET system is very slow, you should increase the equilibration time. The

applied potential should be the starting potential of the potential sweep. These values should be entered in mV and ms followed by an m, for example, 0.15 V should be entered as 150m.

```

43 cell_on
44 #equilibrate at -0.5 V for 5 seconds, using a CA measurement
45 #meas_loop_ca potential(V) current_WE0(A) start(V) interval_time(s) duration(s) poly_we(1 b)

```

```

46 meas_loop_ca p c -10m 20m 1000m poly_we(1 b)
47 #selects ch 1 as the active channel
48 set pstat chan 1

```

Figure 8 lines 43 to 48 for equilibration of the MethodSCRIPT

Lines 47 to 53 set the right current for the working electrode of the bipot channel (WE1) based on the values recorded during the measurement in line 46.

Lines 58 to 85 are the actual measurement, i.e. the currents of both electrodes are recorded while the potential on one channel is swept linearly. This linear sweep voltammetry is defined in line 58 (see Figure 9).

```

55 #LSV measurement using channel 0 as WE1 and channel 1 as WE2
56 #WE2 current is stored in var b
57 #meas_loop_lsv potential(V) current_WE0(A) start(V) end(V) step(V) scan_rate(V/s) poly_we(1 current_WE1(A))

```

```

58 meas_loop_lsv p c -10m 1000m 1m 100m poly_we(1 b)
59 #Send package containing set potential and measured WE current.
60 pck_start
61 #The variable 'p' is added to the data packet. The first variable becomes X axis in PStrace
62 pck add p

```

Figure 9 lines 55 to 62 for the potential sweep in the MethodSCRIPT

There you define where the sweep relative to the RE0 will start and end, how often a value is recorded, and how fast the potential changes.

Lines 59 to 68 make sure the recorded data is sent to the computer, which sends the MethodSCRIPT code to the EmStat Pico.

Lines 77 to 84 perform the current ranging for the bipot channel (WE1).

After the script is finished the cell will be turned off.

2.4 How to perform the MethodSCRIPT

Close the editor by clicking on the X. The technique in the dropdown menu should be now *MethodSCRIPT Sandbox*. You just need to press the Run-button (green triangle) to start the measurement. Two curves will be added to the graph. The applied potential is the x-axes and the current of the first WE (WE0, channel 0) and the second WE (WE1, channel 1) are the y-axes.

2.5 Example of ISFET Transfer Curve

A pH-sensitive ion-selective field effect transistor (ISFET) and Ag/AgCL reference electrode by Winsense (Bangkok, Thailand) was immersed in solutions with different pH values and the transfer curves were measured with the EmStat Pico. The standard buffer solutions of pH 4.0, pH 7.0, and pH 10.0 were prepared by dissolving the powders in distilled water to get a final volume of 500 mL. The powders are the Hydrion Tri-Chek Buffer Chemvelope purchased from Micro Essential Laboratory (Brooklyn, NY, USA). The

gate potential V_G was scanned from -2 V to +250 mV with a scan rate of 100 mV/s and a step potential of 1 mV. The source-drain potential V_{SD} was a constant 300 mV.

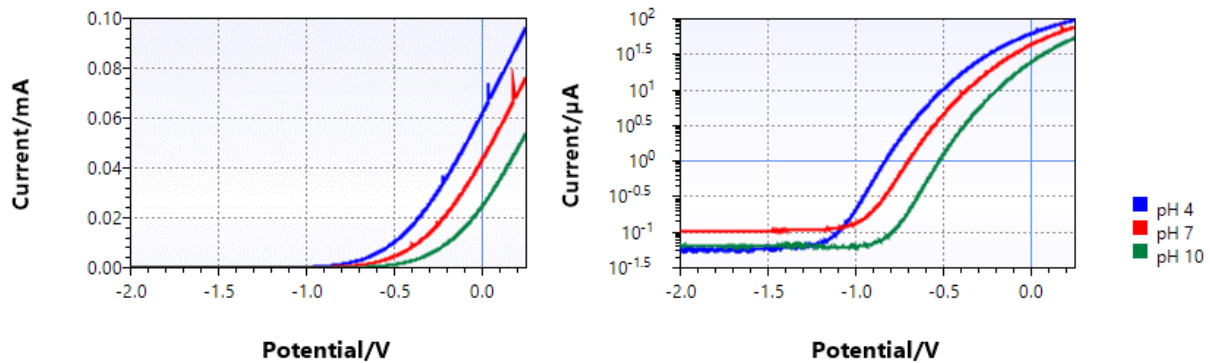


Figure 10 Transfer curves of a pH ISFET by Winsense in pH 4, 7, and 10 in linear (left) and logarithmic scale (right)

The resulting plots of I_{SD} versus V_G are shown in linear and logarithmic scales in Figure 10.

With this characterization, the settings for a pH measurement can be determined. In the example above we find that a V_G of 300 mV and a V_{SD} of 500 mV delivers a linear relationship between $\log_{10}(I_{SD})$ and the pH value. After setting these parameters as constant potentials, the ISFET was immersed in a pH 4 solution for 5 minutes, then pH 7 for 5 minutes, followed by pH 10 for 5 minutes. To demonstrate reproducibility pH 7 and pH 4 were measured again. The results in Figure 11 show that the current correlates with the pH value.

This is example is taken from the previous mentioned publication:

<https://chemrxiv.org/engage/chemrxiv/article-details/6284b89f6b12b6bc00760e65>

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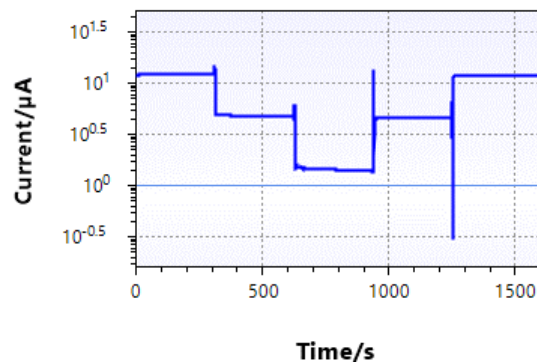


Figure 11 I_{SD} vs time for the pH ISFET, 5 minutes pH 4, 5 minutes pH 7, 5 minutes pH 10, 5 minutes pH 7, 5 minutes pH 4

3 Acknowledgments

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4 Annex

4.1 MethodSCRIPT for FET measurements

```

e
#This sets the device into MethodSCRIPT mode
#-----
#declare variables
# c = WE0 current
var c
# p = set_potential (Vgs for transfer characteristics setup, Vds for output characteristics setup)
var p
# b = WE1 current
var b
#-----
#Set up the current measurement on WE1 (i.e. channel 1 in bipot mode)
#select channel 1
set_pgstat_chan 1
#set channel 1 to poly_we (bipot) mode
set_pgstat_mode 5
#Set required bandwidth (Hz)
set_max_bandwidth 100
#set poly_we (bipot) mode = 0, thus WE1 maintains a fixed voltage relative to RE0
set_poly_we_mode 0
#set starting current range e.g. for 100uA use "set_range ba 100u"
set_range ba 5m
#set WE1 to RE0 offset voltage. (Vds for transfer characteristics setup, Vgs for output characteristics setup)
set_e -20m
#-----
#Set up the current measurement on WE0 (i.e. channel 0 in low-speed mode)
#select channel 0
set_pgstat_chan 0
#set channel 0 to low-speed mode
set_pgstat_mode 2
#Set required bandwidth (Hz)
set_max_bandwidth 100
#Set the range for ch 0 (RE0 to WE0) voltage sweep
#(Vgs for transfer characteristics setup, Vds for output characteristics setup)
set_range_minmax da -1250m 1250m
set_autoranging ba 1n 5m
#set starting current range e.g. for 100uA use "set_range ba 100u"
set_range ba 5m
#-----
#-----
#Perform the voltage scan, reporting RE0 to WE0 potential, WE0 current, and WE1 current
#Turn the cell on, any settings set when the cell was off will be applied here.
cell_on
#equilibrate at -0.5 V for 5 seconds, using a CA measurement
#meas_loop_ca potential(V) current_WE0(A) start(V) interval_time(s) duration(s) poly_we(1 b)
meas_loop_ca p c -10m 20m 1000m poly_we(1 b)
  #selects ch 1 as the active channel
  set_pgstat_chan 1
  #Set the current range corresponding to the measured current on the secondary WE

```



```
mul_var b 1150m
set_range ba b
#selects ch 0 as the active channel
set_pgstat_chan 0
endloop
#LSV measurement using channel 0 as WE1 and channel 1 as WE2
#WE2 current is stored in var b
#meas_loop_lsv potential(V) current_WE0(A) start(V) end(V) step(V) scan_rate(V/s) poly_we(1
current_WE1(A))
meas_loop_lsv p c -10m 1000m 1m 100m poly_we(1 b)
#Send a package containing set potential and measured WE current.
pck_start
#The variable 'p' is added to the data packet. The first variable becomes the x-axis in PStrace
pck_add p
#The variable 'c' is added to the data packet.
pck_add c
#The variable 'b' is added to the data packet.
pck_add b
#Signals the end of a measurement data package.
pck_end
#selects ch 0 as the active channel
#set_pgstat_chan 0
#Measures the WE0 current for 100 ns and stores the value in 'c'
#meas 100n c ba
#Set the current range corresponding to the measured current on the main WE
#//mul_var c 1150m
#set_range ba c
#selects ch 1 as the active channel
set_pgstat_chan 1
#Measures the WE1 current for 100 ns and stores the value in 'b'
#meas 100n b ba
#Set the current range corresponding to the measured current on the secondary WE
mul_var b 1150m
set_range ba b
#selects ch 0 as the active channel
set_pgstat_chan 0
endloop
#Turn cell off when finished or aborted
on_finished:
cell_off
```