

Q-Sense E4 Quartz Crystal Microbalance (QCM-D) Procedure

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The user will need to provide their own sensors. For information and to order sensors go to the Q-Sense website page <http://www.q-sense.com/q-sense-sensors>

Samples can be degassed in a sonicator bath prior to running to help remove air bubbles.

IMPORTANT: Make sure the sample fluid is chemically compatible with the measurement chamber and the sensor.

Below is a list of materials of the QCM-D.

Chamber walls: Titanium grade 2

Temperature stabilizing loop: Viton rubber

O-ring: Viton rubber

Tubing: Teflon

Pump Tubing: Tygon ST

I. Facilities Billing System (FBS)



To Schedule time and to use the instrument computer a FBS account is required. After training you will receive an invite for FBS or if you already have a FBS account the QCM-D calendar will be added to it.

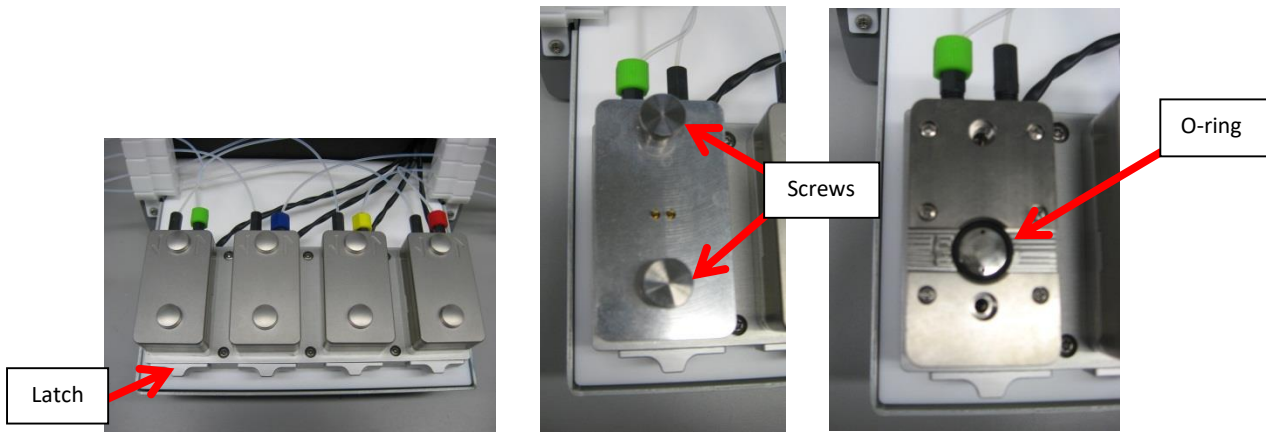


The instrument time is billed through FBS. Your FBS scheduled time + any extra = your recharged time. Recharge is calculated at an hourly rate with a minimum charge of 1 hour. For current recharge rates go to the MRL website.

- ❖ To begin an instrument session, you must log onto FBS first, and either click on **Start Timer** if a reservation has already been made or select **Walk-Up**
To do so use the FBS designated computer in the lab or any internet connected device, and navigate to <http://ucsb.fbs.io>
- ❖ Now you can log onto your account on the instrument computer. Remember to log off your computer account when finished.
- ❖ Once the session is finished you must log into your FBS account and click **Stop Timer**. This will stop your FBS time. If you do not do this you could incur extra charges.
- ❖ The paper log sheet by the instrument is used as back up for the FBS system. Remember to mark your time, recharge number, and any notes or problems you would like to convey, feel free to use as many lines on the page as needed to be clear.
- ⚠ No shows will be charged **75%** of the scheduled time. If you cannot use your reserved time cancel it.
- ⚠ FBS records the billable time as the longer time between the scheduled time and the time used. So if your scheduled time is longer than the actual time on the instrument then the scheduled time will be charged.

II. Inserting the sensor

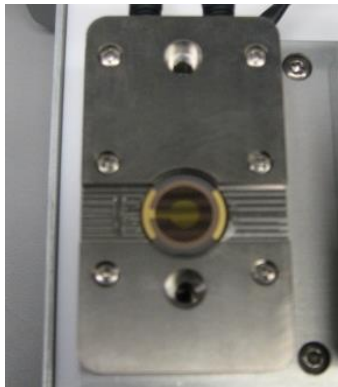
- 1) Open the lid of the chamber platform. Slide the latch to unlock the flow module, lift the module off the chamber platform. For stability flip the module upside down and place it on the platform. Unscrew the flow module. Take care not to touch anything in the module with your fingers. Once open make sure the o-ring is lying flat.



- 2) Carefully take the sensor holding it on the outside ring, avoiding the electrodes, with forceps.



- 3) Place the sensor so it is active side down, resting on the o-ring. It will look like this when aligned correctly.



- 4) Screw the contact block onto the module. **DO NOT OVER TIGHTEN** the sensors are very delicate and can break easily. Place the flow module back on to the chamber platform holding it firmly to the left while the latch is closed.


*Make sure the right tubing with the corresponding module is set up for the Milli-Q water and sample waste (waste tubing is color coded).

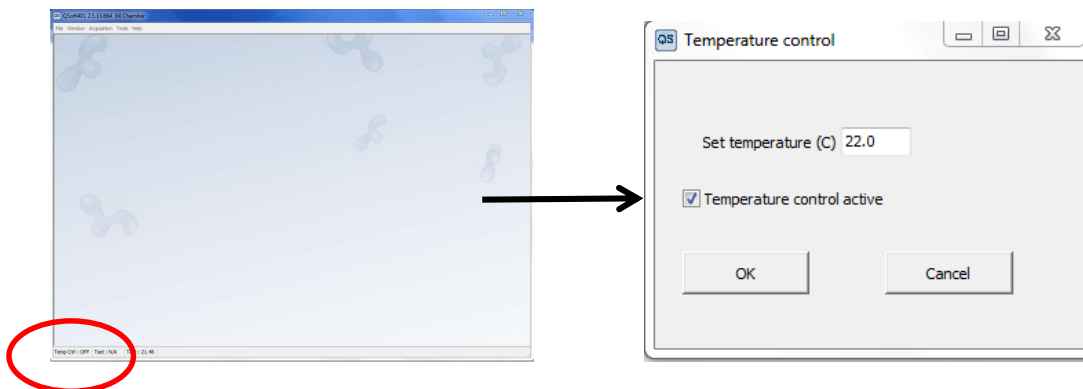
III. Preparing for a measurement

- 1) Write your information, including recharge number on the paper log sheet, start your FBS timer, and log on to the instrument computer.

- 2) Turn on the electronics unit by pressing the silver button on the front (will light up green when on).

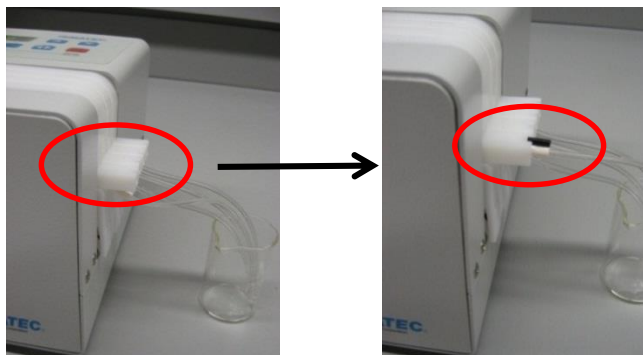


- 3) Open up QSoft401  and turn on the temperature controller by clicking the control panel on the bottom left. Set to 22 degrees C and check the temperature control active box, click OK.



If a higher temperature (up to 65 degrees C) is desired the samples must be heated first before entering the QCM by placing them on a heating stage which the user will need to provide. Allow 5-10 minutes for equilibration, longer if the set point differs greatly from room temperature. Heat the samples to slightly above the desired temperature (5-10) degrees to account for any cooling taking place as the sample travels to the module.

Turn the pump on (switch is on the back). The tubing on the waste side should be unhooked from the cassette when not in use. Pull the waste side tubing tight and place it in the corresponding groove.

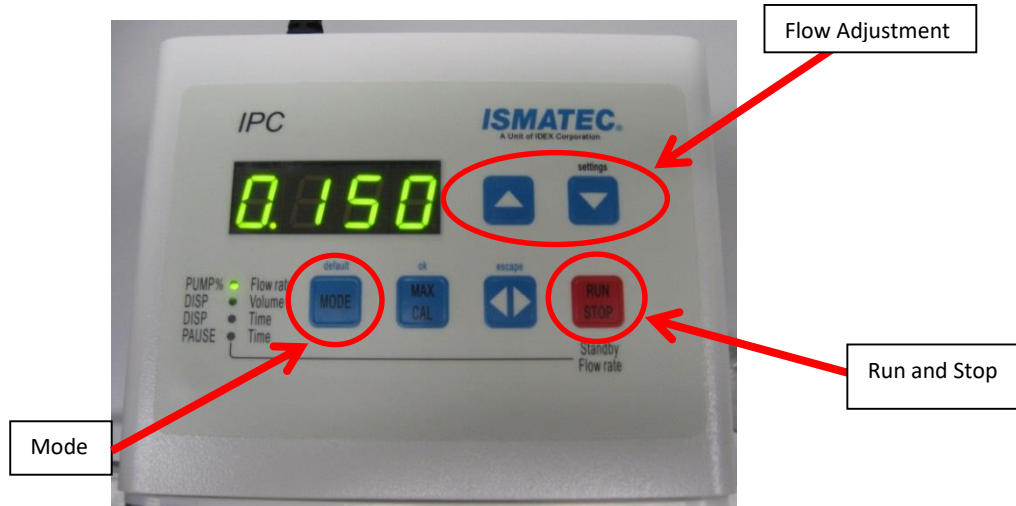


Have the input tubing already in Milli-Q water or a buffer solution before starting the pump. To change between liquids stop the pump first. This will reduce the chances of air getting in. The default

setting is **Pump%** which is uL/min or ml/min with a pump rate of **150 uL/min**. The flow can be adjusted using the up and down arrows. **Do Not** exceed 1 ml/min as this will not allow enough time for tepearture stabilizaiton. If volume or time mode is desired press the **mode** button to change. For more information refer to the ISMATEC user’s manual. If any of the settings are changed they must be set back to the default settings at the end of machine use.

NOTE: Sometimes it is desirable to find your resonances and run a baseline in air. If this is the case the tubing does not have to be in Milli-Q water or a buffer to start.

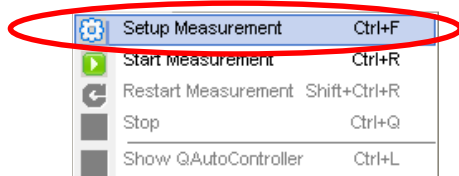
- 4) Start the pump by pressing the **Run Stop** button.



- 5) Run the pump and watch to make sure the liquid is flowing all the way through the module and there are no air bubbles present.

IV. Running a measurement

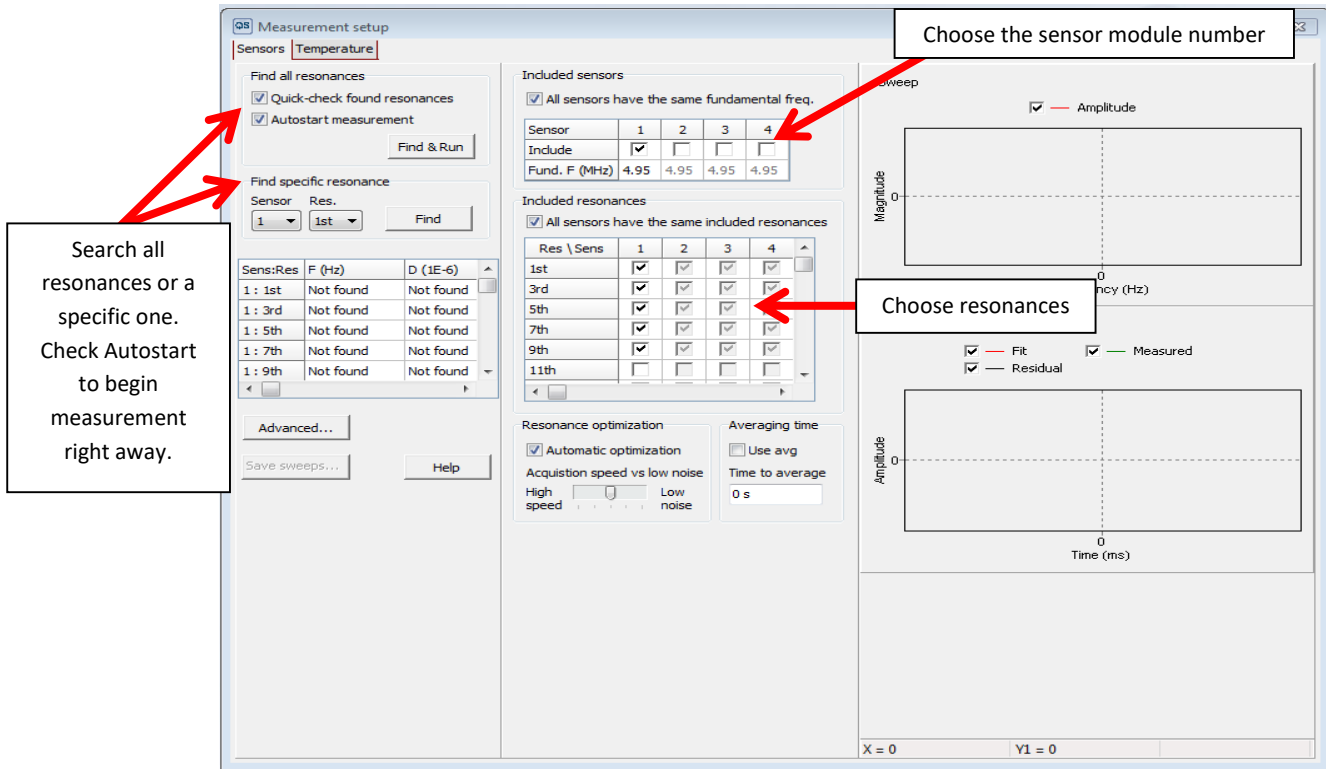
- 1) Before running a measurement the resonance frequencies of the sensors must first be found. In the toolbar go to **Acquisition → Setup Measurement**



Select which modules are in use (1-4). To find the resonance of a sensor, click **Find all resonances** or **Find specific resonance**. It is generally recommended for a run that all available resonances should be used.

A sweep chart will appear where the resonant amplitude versus frequency is shown. QSoft will use the peak with the largest deviation from the mean of the sweep and use this frequency as the starting value, f1. The electronics unit will then excite the crystal with a drive signal with frequency f1. After the signal is turned off a decay curve is acquired and QSoft calculates the measured f and D values from the sensor’s decay curve by doing a numerical fit.

Sweep curves can be saved by clicking the save sweeps button.



Under the **Advanced** button there are additional resonance settings. Usually these do not need to be altered.

Most users will use the Automatic optimization. For specific adjustments uncheck the box to access the following parameter options:

Resonance Optimization: If very fast processes need to be followed a higher amount of data points per minute are sampled by moving the pin towards High speed. The trade off is higher signal to noise ratio.

For most conditions it is recommended to use the standard settings.

Averaging time: If a very long measurement (many hours) is done and slow kinetics are expected check this box. QSoft will collect f and D data as normal during the time specified in the time to average edit box. When this time is up, QSoft will average all value data points during the averaging time and store the averaged values. This is done individually for each resonance.

*If there appear to be problems finding resonances it is possible to individually adjust the settings for each resonance by unselecting the automatically optimize all resonances checkbox.

Center Frequency(MHz): Sets the median frequency value around which the resonance search should be done.

Frequency range (kHz): Sets how large the frequency interval around “Center Frequency” should be.

Drive Amplitude: Determines the amplification of the drive amplifier, indirectly how much voltage should be applied over the sensor to excite it into oscillation. Normally 0.5-1.0 is enough.

Drive Time (ms): Sets how long the sensor should be excited before the voltage is turned off and the decay curve is recorded. This seldom needs to be changed.

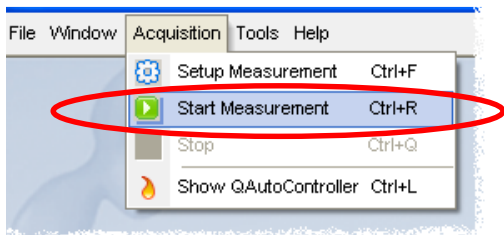
Decay amplification: Sets the amplification level of the signal from the sensor before it reaches the analog to digital converter.

Decay Length: Sets how many data points should be collected for the numerical fit.

Decay Average: Sets how many decay curve fittings should be calculated before presenting an averaged value. 16 is the default setting. Lower values will give higher time resolution but also more noise.

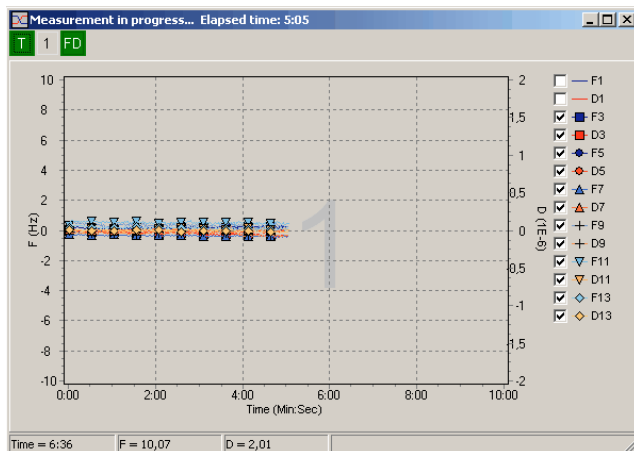
Decay sample rate (MHz): Sets how detailed the data point collection should be for fitting of the decay curve. For air measurements around 0.5 is recommended, for water measurements a value around 2 is recommended. Since the decay is much quicker in water than in air, more data points need to be collected before it has leveled out, and a higher sampling frequency is needed.

- 2) Before a sample is run it is recommended that a baseline is run. This may be done with Milli-Q water. Go to **Acquisition** → **Start Measurement**. One f and D vs. Time chart will be shown for each sensor chosen (1-4).



NOTE: Another option is to check the Autostart Measurement box in Setup Measurement, this will begin measurement right after the resonances have been found.

Run for about 5 minutes. The baseline should be flat. For example a standard Au sensor in water should not drift more than 2 Hz/hour. **Note: If you run a baseline in air the drift will be larger.**



- 3) Once a baseline is established. Stop the pump and then switch the tube to the sample or buffer container. Start the pump. Watch for bubbles. After stabilization and desired time stop the

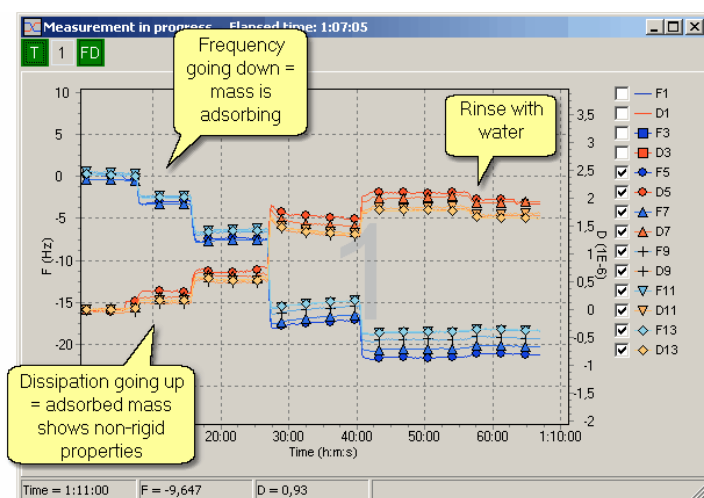
pump and switch to the next sample of choice. Continue like this until the desired multilayer is achieved.

A shift between buffers will effect the measurement. This will show up as an instantaneous change which is reversible and surface dependent. The best way to avoid this is to keep the concentration of the analytes (e.g. proteins) down and to use the same buffer as much as possible throughout the experiment. Also you can do a final rinse with the baseline buffer after a completed measurement you will be certain that no buffer effect will disturb the interpretation.

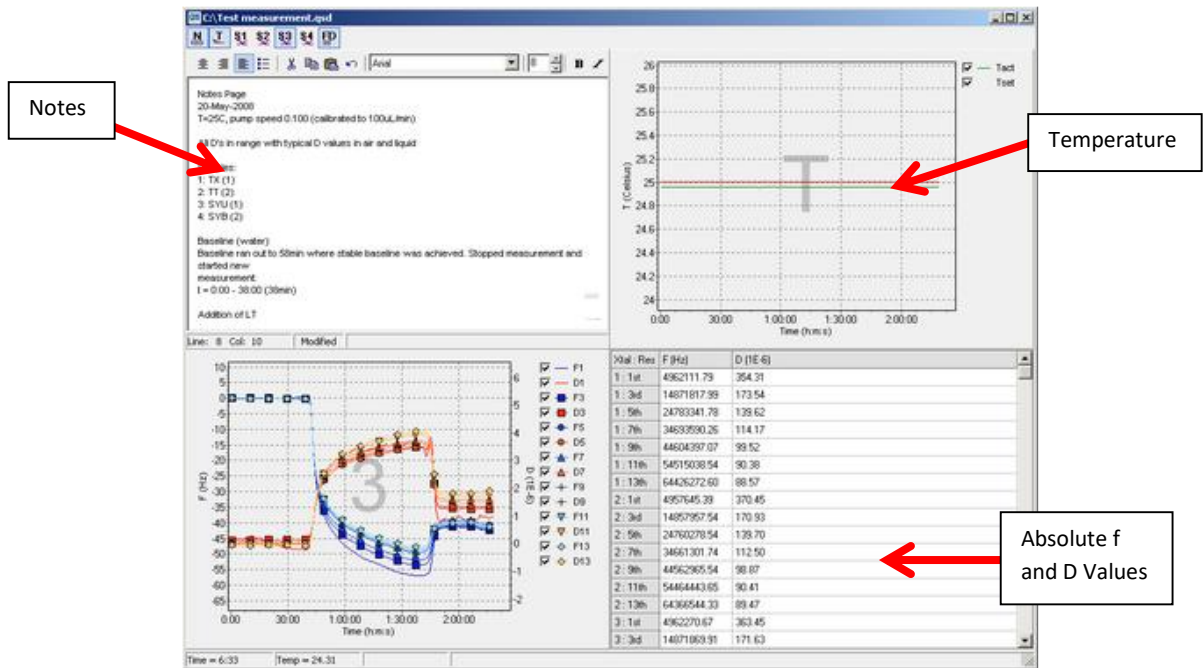
*****NOTE: If bubbles continue for a long time or you do not see flow there is a problem.**

Possibilities include, the sensor may have cracked, the liquid was not properly degassed or there is a problem with the tubing.

The finished run may look something like this.



There are extra panels available; notes (text editor) there is a time stamp option in the tool bar for noting the time of an event, a temperature chart and a table with absolute values for f and D. These panels can be controlled with the buttons in the tool bar. Double clicking on a panel will show that panel and hide the rest.

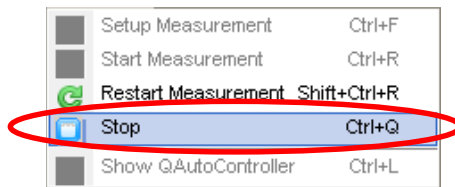


Right clicking gives a quick menu for hiding or showing f and D values (this is also achieved by clicking or unclicking the corresponding boxes on the measurement panel). The default offset is 0 for f and D. To change the offset click on the Offset option.

To Zoom in on an area hold the left mouse button and drag down and to the right. To Un Zoom drag up and to the left.

V. Finishing up and data saving

- 1) To stop acquisition go to **Acquisition** → **Stop**



A Save file window will appear. Enter a file name and click Save (**this saves in the Q-Soft format**). If the file is very large from a long measurement (several hours) the condense function can be used to make data handling easier. Go to **File** → **Condense**.



To save as a text file open your file in D-Find

- 2) Make sure to rinse the module(s) and tubing with Milli-Q water pumping for at least 10 minutes after the last sample or buffer has been run. To do some extra strong flushes hold down the Max Cal button on the pump. When done rinsing remove the tubing from the water and run air about 5 minutes to dry the tubing
- 3) Turn off the Temperature Control.

- 4) Open the module(s) used and remove the sensor(s). Clean the modules with Milli-Q water and/or ethanol and dry completely with nitrogen gas. If liquid is left it can corrode the modules. Sensors can be rinsed and dried in the same way. Depending on the sensor/sample type a different cleaning procedure may be needed. Please see the manual for more information.
- 5) Unhook the tubing on the waste side of the pump to relieve the tension.
- 6) If any of the pump settings have been changed set them back to the default settings.
- 7) Logoff the computer and turn off the QCM electronics.
- 8) Make sure to write down the log off time on the log sheet.
- 9) Go to FBS and click “Stop Timer”, make sure to log out of your FBS account.
- 10) Clean up waste and other items.