RheoSense, Inc.

m-VROC
(Viscometer/Rheometer-On-a-Chip)
User's Manual

900003.5-mVROC_User's_Manual

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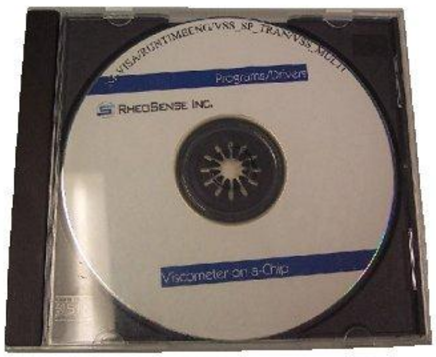
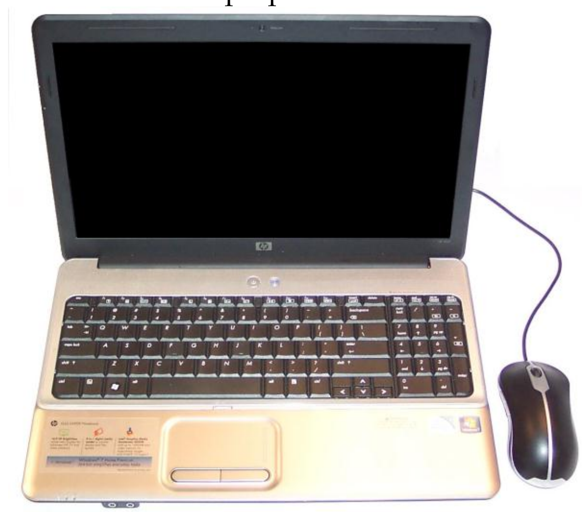
1. Unpacking

Check the package for all the following components.

software CD: OR laptop:





m-VROC unit:

USB cord:

VROC Cell assembly:

power cord for the *m*-VROC unit:

water tubing:

document packet:

Aquet detergent:

plastic needles, unions, and fuses:

If the Thermocube water bath has been purchased with the system, also check for the following components.

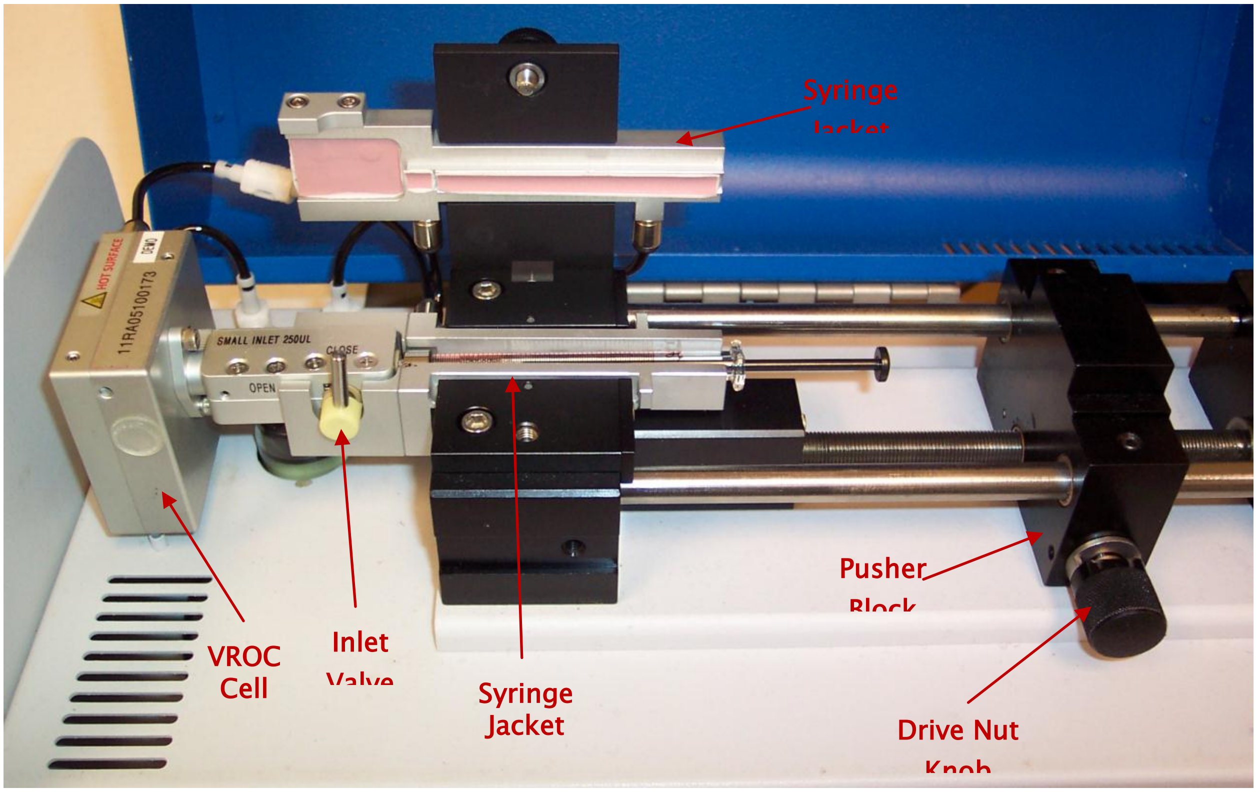
Thermocube unit:

USB to serial adapter:

fluid connection hose assembly:

power cord for the Thermocube unit:

Parts of the *m*-VROC unit:



2. *m*-VROC Specifications*

- Viscosity measurement accuracy: +/- 2.5% of full scale or +/- 5% of reading, whichever is smaller
- Repeatability: +/- 0.5%
- Temperature accuracy: +/- 0.1 °C
- Viscosity range (depending on VROC Cell type*): 0.2 – 1,000,000 mPa-s
- Shear rate range (depending on VROC Cell type*): 0.3 – 1,100,000 1/s
- Minimum sample volume: <50 µl
- Available syringe sizes: 100 µl, 250 µl, 500 µl, 1 ml, 10 ml
- Dimensions: 10″ L x 15.5″ W x 7 H
- Weight: 16 lbs
- Power Consumption: 60 W
- Ambient temperature operating range: 20 – 27 °C
- Ambient humidity operating range: 0 – 60%
- Purge air supply flow (optional): 20 – 40 scfh (10-20 slpm) at 5-10 psig
- Purge air supply condition: < 1 °C dew point
- Heating/cooling fluid supply (optional): 5 slpm at 30 psig
- Temperature measurement range: 18 – 50 °C (4 – 80 °C optional)
- Wetted materials: borosilicate glass, silicon, PTFE, ETFE, PEEK, gold, Perlast (Kalrez optional)
- CE certified
- Temperature control performance with optional water bath circulator (Thermocube):
  - Range: 4 – 68 °C with 100 µl, 250 µl, 500 µl, and 1000 µl syringe jackets
  - Range: 4 – 65 °C with 10 ml syringe jacket
  - Stability: +/- 0.075 °C

- Ramping rate and settling time: 6 °C/minute plus 3 minutes when heating, 4 °C/minute plus 3 minutes when cooling

* RheoSense, Inc. reserves the right to change specifications without notice.

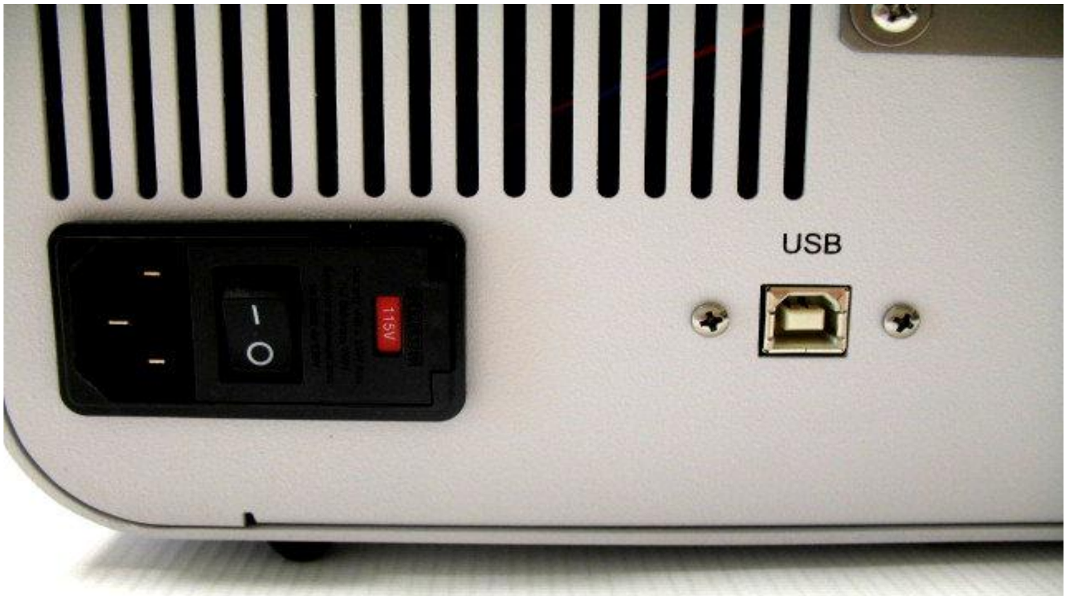
3. Precautions

1. **Always** use the provided software package with the VROC Cell. The program has built-in safety features that prevent the VROC Cell from being overloaded.
2. Only syringes from the Hamilton Company can be used in the *m*-VROC unit. These may be purchased from RheoSense or from the manufacturer. Check with a RheoSense representative for compatible syringe sizes.
3. **Always** clean the VROC Cell thoroughly after each use, and before storing the VROC Cell. Please refer to section 7a for cleaning procedures.
4. Particles larger than 10% of the flow channel gap should be filtered out of the sample to avoid clogging the VROC Cell (for example, if using the A-02 VROC Cell, particles in the sample should be no larger than 2 μm).
5. Keep the inside of the tubes and the flow channel of the VROC Cell wet **at all times**. Dried sample may clog the flow channel and cause irreversible damage to the VROC Cell. See section 7d (Storing the VROC Cell) for more details.
6. Keep the inlet valve closed (small arm in the vertical position) when not using the VROC Cell.
7. **Never** test immiscible samples back to back. If two immiscible liquids are introduced into the VROC Cell, permanent damage may result. See section 8d (Testing Immiscible Samples) for more details.
8. **Always** leave the waste tube connected to the VROC Cell assembly. Removing this tube exposes the sensor area inside the VROC Cell, which may allow the sample to dry in the flow channel causing damage or clogging.
9. **Always** operate the system with the VROC Cell assembly properly positioned (see the pictures in section 5b).
10. If the pusher block becomes jammed during operation, use the clear stall button in the software to unjam it. Do not attempt to unjam it manually.
11. If using a water bath circulator, use only distilled water and/or cooling fluids such as ethylene glycol.

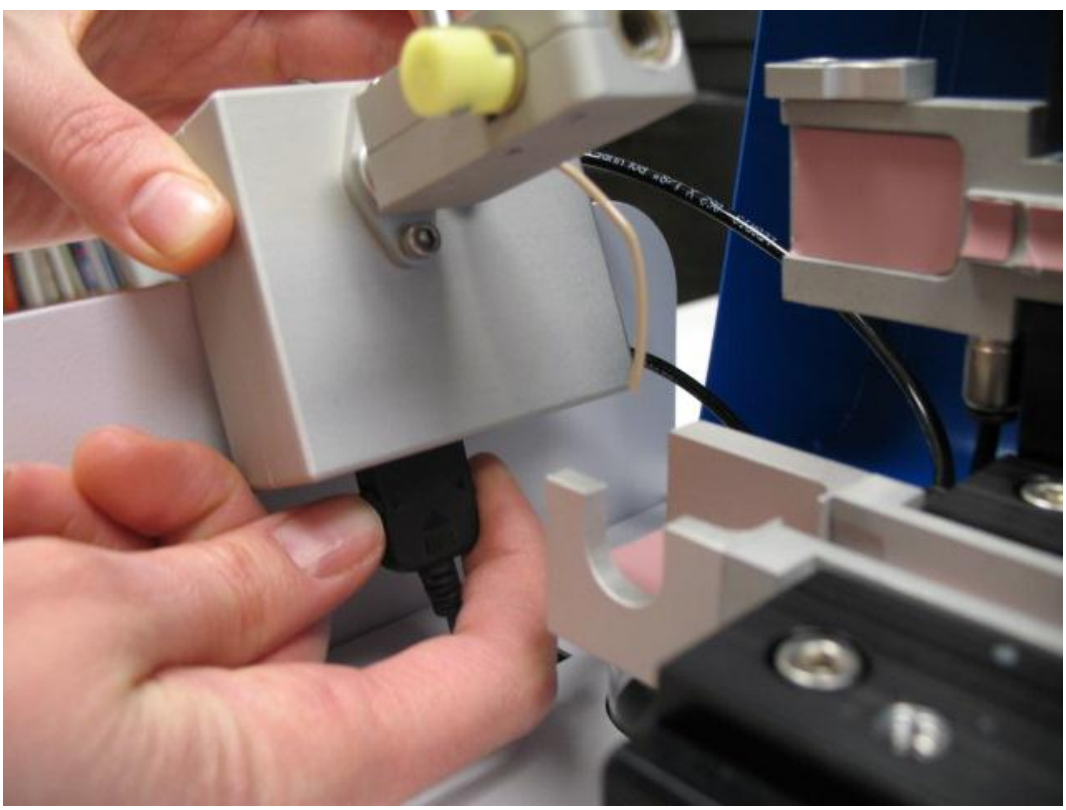
4. System Setup

A. *m*-VROC Unit Setup:

1. Connect the USB cord and power cord to the rear panel of the *m*-VROC unit:



2. DO NOT connect the other end of the USB cord to the computer until the USB driver has been installed (See the *m*-VROC Control Software Installation Guide). Plug the *m*-VROC unit's power cord into a power source. Leave the power switch on the back of the *m*-VROC unit off.
3. Connect the bottom of the VROC Cell assembly to the plug from the base of the *m*-VROC unit (Note that the plug only connects one way. If connecting the plug to the assembly is difficult, rotate the plug 180° and try again.):



B. Temperature Control Hardware Setup

The temperature control jacket (housing the VROC Cell and syringe) can be operated at temperatures between 4 °C and 80 °C (between 4 °C and 68 °C with fully automated control). This allows for testing of a sample at a constant temperature within this range. The *m*-VROC system has two levels of temperature control: fully automated (closed loop) and partially automated (open loop). All *m*-VROC models come with the partial temperature control feature. On *m*-VROC models that are equipped with the full temperature control feature, partial temperature control may also be used if desired. Any model of water bath circulator may be used to partially automate the temperature control. For fully automated temperature control, a USB-to-serial adapter and a Thermocube water bath circulator will be needed.

Use only distilled water and/or cooling fluids such as ethylene glycol in the water bath circulator as recommended by the manufacturer.

Make sure to use a dry air supply with 1/4” outer diameter tubing if temperatures below room temperature are desired. This will prevent condensation from reaching the sensor. Set the air flow between 20 and 40 cubic feet/hour (between 10 and 20 liters/minute). The dew point of the dry air must be 0 °C or lower.

If partially automated temperature control is desired:

1. Follow all the steps in section 4a.
2. Connect a water bath and/or a dry air supply to the ports on the back panel of the *m*-VROC unit (RheoSense provides the mating parts of the connectors, which may be attached to the tubes from the water bath circulator by pushing each tube over the barb inside the back of the connector. If the optional Thermocube chiller was purchased, appropriate hoses are included to connect the chiller to the *m*-VROC unit.):

3. The best place to position the water bath circulator is on the same level and to the right of the *m*-VROC unit. This will allow the fan in the water bath unit to blow air away from the *m*-VROC unit. To achieve the specified performance, leave at least 6" of clearance around the water bath. If the water bath cannot be placed beside the *m*-VROC unit, place it as close as possible and on the same level as the *m*-VROC unit. Do not extend the water lines to more than 8'.
4. Turn on the water bath circulator and set the bath to circulate between 0.1 and 0.5 liters/minute (0.25 to 0.125 gallons/minute).
5. If testing below 18 °C, begin the dry air purge.
6. Turn on the *m*-VROC unit.
7. Follow the instructions in section 5 for loading a sample.
8. After a sample is loaded, close the cover of the *m*-VROC unit and open the *m*-VROC control software. Fill in the appropriate information in the Measurement Setup tab, and then go to the Temperature Control/Measurement Advisor tab (see section 6a-iv) to make sure the water bath is holding the temperature steady at the desired level before using the other parts of the software. There will be a slight temperature gradient between the water bath set point and the measured temperature inside the sensor. For instance, if 5 °C operation is desired, try setting the water bath to 2 °C (set the temperature of the water bath higher than the target temperature if temperatures above room temperature are desired), and monitor the temperature of the sensor. It should take between 3 and 6 minutes for the temperature of the sensor and fluid in the syringe to come to equilibrium once the bath has reached the desired temperature. Please see section 8a for additional information on achieving thermal equilibrium.

If fully automated temperature control is desired:

1. Follow all the steps in section 4a.
2. Use the fluid connection hose assembly to connect the Thermocube water bath circulator to the *m*-VROC unit, making sure to match the hose label with the port label (Note that the connectors that attach to the *m*-VROC unit can only be connected in one way.):

3. Use the USB to serial adapter to connect the Thermocube to one of the control computer's USB ports:

4. The best place to position the Thermocube is on the same level and to the right of the *m*-VROC unit. This will allow the fan in the Thermocube to blow air away from the *m*-VROC unit. To achieve the specified performance, leave at least 6" of clearance around the Thermocube. If the Thermocube cannot be placed beside the *m*-VROC unit, place it as close as possible and on the same level as the *m*-VROC unit. Do not extend the water lines to more than 8'.
5. Make sure the fluid connection hose assembly is ALWAYS connected to the Thermocube when in operation. The fluid connection hose assembly includes a bypass segment which protects the pump of the Thermocube. Never disconnect the bypass segment from the fluid connection hose assembly. Poor performance and/or pump damage could result if the bypass tube is disconnected.
6. Turn on the water bath circulator.
7. It is recommended not to change the default value of 25 °C on the water bath circulator. Use the Temperature Control/Measurement Advisor tab in the software to do this. See section 6 for instructions on using the temperature control portion of the software.

5. Sample Loading

Good sample loading technique is important for accurate viscosity measurements. Bubbles, contaminants, etc. will adversely affect measurement results. Refer to section 8 for tips on sample loading.

A. Loading Sample into the Syringe

For most sample fluids, the best method for loading is:

1. Use the plastic intake tube (plastic needle) and union (included with the syringe) to load the sample solution into the syringe:

2. Air bubbles can be generated during sample loading. Look at the fluid in the syringe to verify that all visible bubbles are at the top. Dislodge any bubbles stuck to the plunger or syringe wall by gently tapping the syringe. Then, push the plunger in until a small amount of sample is expelled from the end of the plastic intake tube:

3. If air bubbles are not removed, they can become trapped inside the flow channel of the VROC Cell, causing non-linear pressure profiles due to distorted flow fields. Their presence will also delay the response time, and be a source of error in viscosity measurement as the bubbles are compressed when pumping starts.

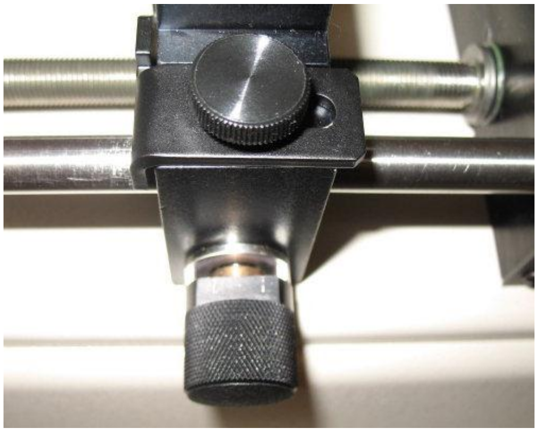
Please see section 8c (Advanced Measurement Techniques) for special instructions on loading high viscosity samples.

B. Loading the Syringe on the Pump

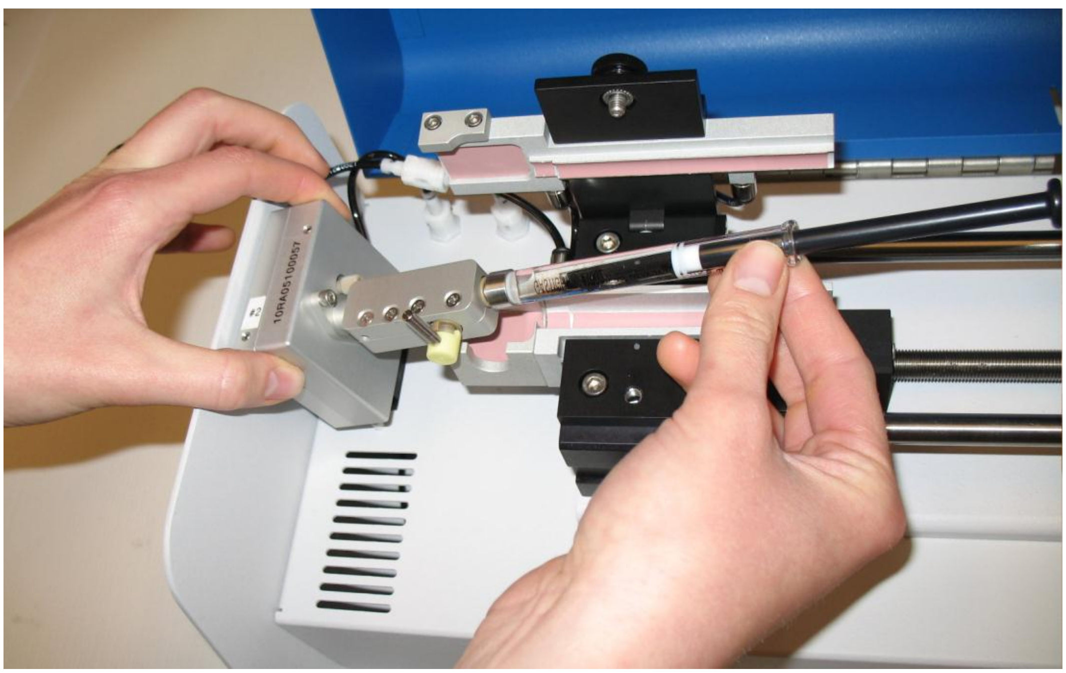
1. Begin by making sure the drive nut knob is unlocked. It is located on the side of the pusher block (where the end of the plunger will rest when the syringe is mounted). If the knob can be pulled straight out (a bit of force is necessary to pull it), it is in the unlocked position. If it can only be turned, it is in the locked position, and a gap will be seen between the nut and the side of the pusher block (a brass rod will be visible in the gap). To unlock the drive nut, turn the knob until it retracts slightly into the pusher block. Slide the pusher block all the way to the right.

drive nut in locked position:

drive nut in unlocked position:

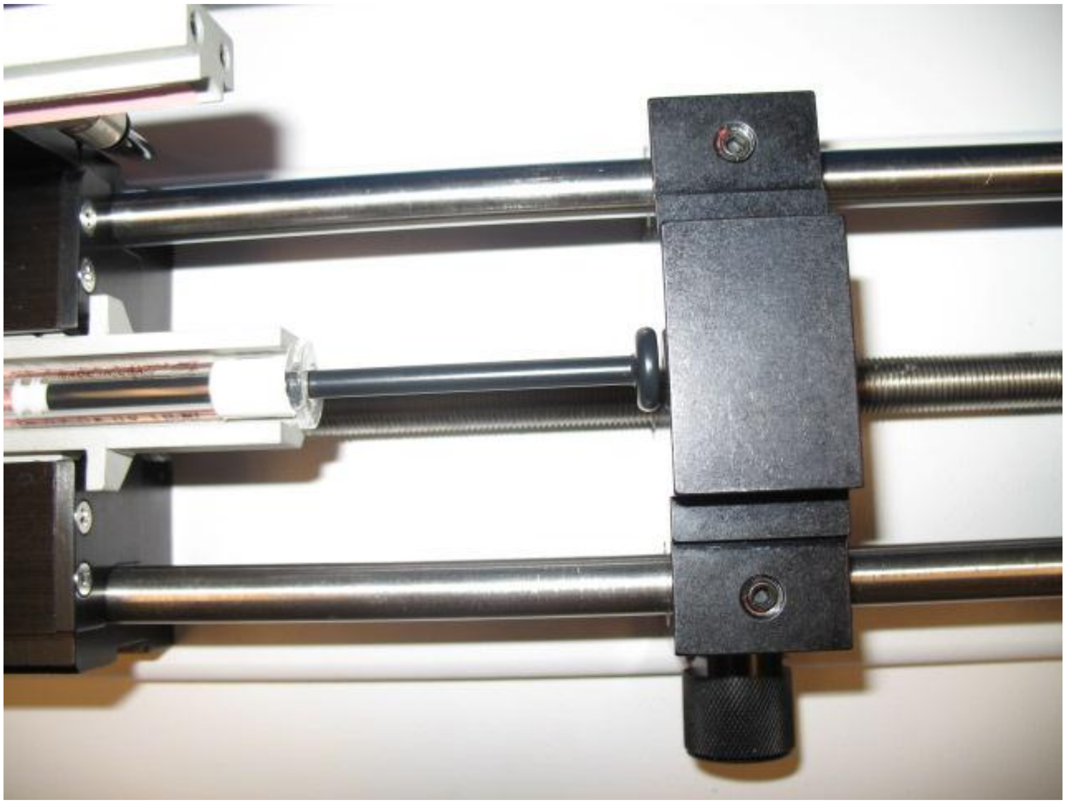


2. Remove the plastic intake tube and union by unscrewing them (removing them as a single unit will reduce dripping) from the end of the syringe.
3. Screw the end of the syringe into the valve block on the VROC Cell assembly, grasping the end of the syringe body (near the plunger entrance point). Finger tightening is sufficient. Make sure the valve to the VROC Cell is closed (the small arm on the side will be vertical). Place the syringe in the bottom of the heater block, making sure the hinge of the valve seats in the cutout properly:

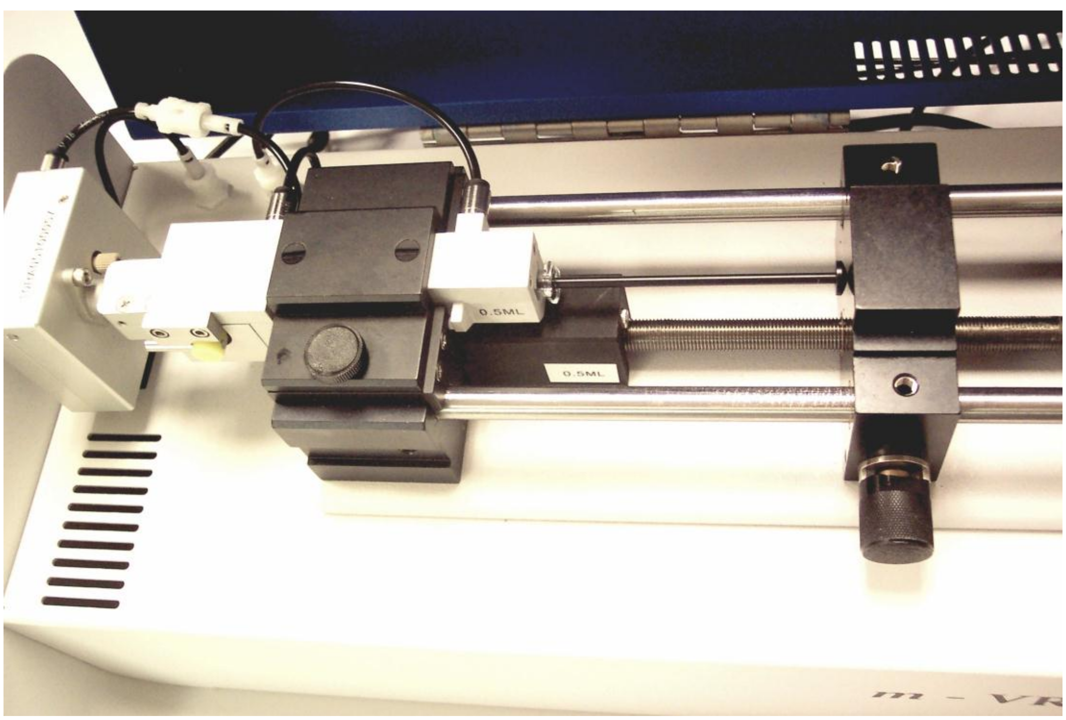


4. Slide the pusher block along the guide rods until the block is close (about 2 mm) to

the end of the syringe plunger, then lock the drive nut by pulling it out and rotating it:



5. Open the VROC Cell valve by rotating the small arm a quarter turn counter-clockwise. Close the lid of the heater block over the syringe, and tighten the thumb screw. When the syringe is properly loaded, it will look like this:



6. The *m*-VROC is now ready to take a viscosity measurement.

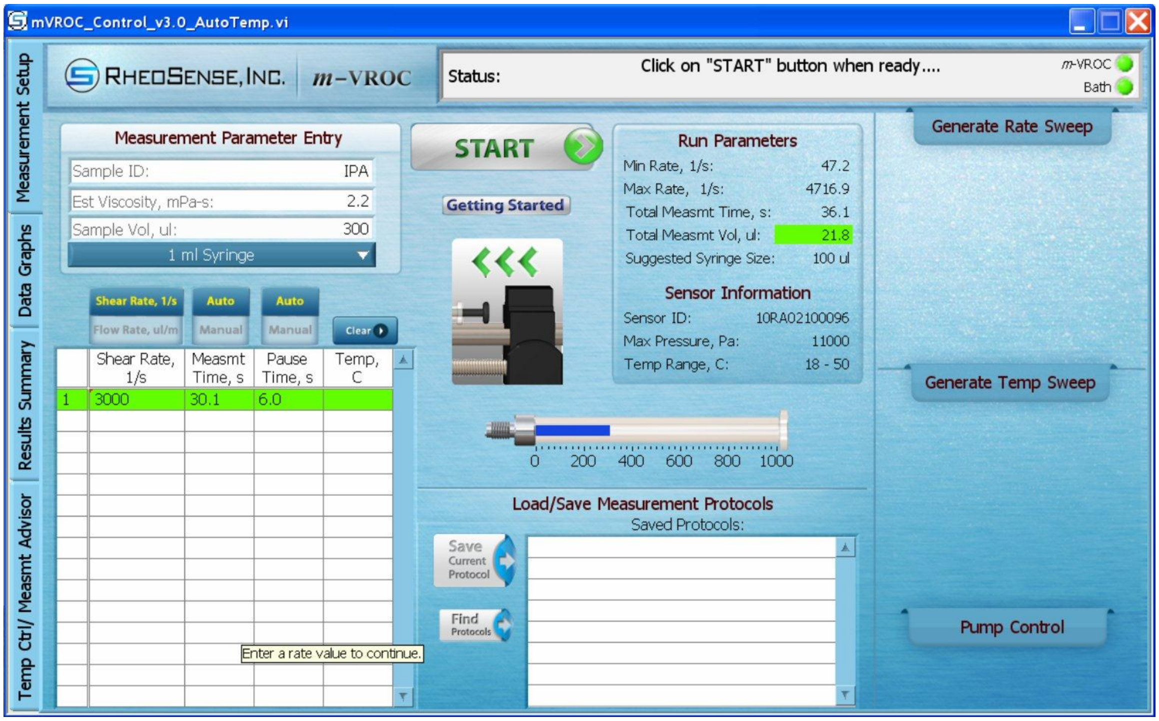
It is important to note that handling the syringe barehanded may affect measurements. Due to the high sensitivity of *m*-VROC, it is necessary to be aware of the temperatures of various components in the system while using it. This is true even if the system is being used in temperature control mode. Holding the syringe barehanded while loading can raise the temperature of the sample enough that a measurement taken shortly after that will be incorrect. This effect is dependent upon the viscosity of the sample, and is more noticeable with lower viscosity samples. This problem can be avoided by either waiting for a few (at least 5) minutes after loading the syringe so the sample has time to return to ambient temperature, or by wearing insulating gloves while loading the sample and mounting the syringe.

6. Taking Measurements With the *m*-VROC Control Software

A. Software Interface Tabs

i. Measurement Setup Tab

The front panel (user interface) looks like this:



- **Status Bar:** This provides instructions on what to do next and also displays what the instrument and/or software is doing. The "*m*-VROC" light is green when the *m*-VROC unit is properly connected and powered on. The "Bath" light will appear green and blinking when the Thermocube water bath circulator is correctly connected and on. The "Bath" light will appear solid green when the temperature has reached a set point and is stable.

Measurement Parameter Entry Area

- **Sample ID:** This is the identification tag of the sample being tested. The sample ID, along with the measurement time stamp, is used as the default data file name when the measurement is completed.
- **Est Viscosity, mPa-s:** The estimated viscosity is used to calculate the minimum and maximum shear or flow rates for the VROC Cell. If the viscosity is not known, try to enter a guess within an order of magnitude of the viscosity and pick a shear or flow rate that is in the middle of the displayed rate range (shown under "Run Parameters"). Enter the chosen rate in the measurement input table and leave "Measmt Time" and "Pause Time" set to "Auto". After the run, update the estimated viscosity ("Est Viscosity" under "Measurement Parameter Entry") with the new value to get better estimates of the minimum and maximum rates available.

- **Sample Vol, ul:** Enter the amount of sample that is in the syringe when it is first loaded. The program keeps track of the remaining volume as the measurement proceeds. Note that it is better to underestimate the amount of sample than overestimate it.
- **Select Syringe Size:** Use this pull-down menu to select the size of the syringe loaded into the *m*-VROC unit.
- **Start:** Clicking on this button begins a measurement.
- **Stop:** Clicking on this button halts a measurement at any point after clicking "Start". This button will appear in place of the "Start" button after "Start" has been clicked.
- **Getting Started:** Click on this button to see general information about using the program.
- **move block to plunger icon:** Click on this to move the pusher block close to the end of the syringe plunger. The pusher block will stop automatically when sample movement is detected in the VROC Cell.

Measurement Input Table

This table appears on the left side of the Measurement Setup tab. It is used to define the measurement parameters of each segment. In most cases, only a rate needs to be entered (if the optional Thermocube water bath is used, a temperature also needs to be entered). Use the 'Generate Rate Sweep' and 'Generate Temperature Sweep' functions to automatically populate this table. If there is not enough sample to complete all the segments entered, background color for that (or those) segment(s) will be shown in yellow (meaning there may be enough) or red (meaning there is not enough). If the requested rate is outside of the VROC Cell or pump range, the row will be shown in yellow (if the rate is too low) or red (if the rate is too high). Only temperatures within the temperature range of the VROC Cell may be entered.

- **Shear Rate/Flow Rate toggle:** Clicking on this button selects the rate units for the measurement: inverse seconds (1/s) for shear rate, or microliters per minute (µl/min) for flow rate. Both rate types are displayed in the test results summary.
- **Shear Rate, 1/s or Flow Rate, ul**: Enter the desired value(s) for the shear or flow rate for each measurement segment. If you have chosen to create a new rate sweep under "Generate Rate Sweep", this column will be automatically populated.
- **Auto/Manual toggle (Measurement Time):** When this is set to "Auto", the program will automatically select the measurement time for a given rate value. The automatic measurement time is the amount of time it takes for the pump lead screw to make one full revolution at the entered rate.
- **Measmt Time, s:** This is the time needed for the measurement to take place. Note that if "Manual" is selected and the value is set to less than the value that appears when "Auto" is selected, measurement results may be inaccurate.
- **Auto/Manual toggle (Pause Time):** When this is set to "Auto", the program will automatically select the pause time for the segment. The pause time is the time it takes the viscosity reading to reach a steady state value. Higher viscosities and slower pumping rates generally require a longer pause time. The program uses these pause times for the listed pumping flow rate (or corresponding shear rate) ranges:

flow rate (µl/min)	pause time (s)
less than or equal to 5	25
less than or equal to 10	15
less than or equal to 40	6
greater than 40	3

- **Pause Time, s:** This is the time at the beginning of a measurement that is disregarded when the program calculates values. During this time, the pressure in the chip is stabilizing.
- **Temp, C:** This is the desired temperature (in degrees Celsius) at which the measurement will be taken. Note that if no Thermocube water bath is being used (or a different bath is in use), this parameter will be ignored.
- **Fall Time, s:** When taking a measurement using temperature control, the instrument will push a small amount of sample through the VROC Cell before collecting data. This is to minimize any bubbles that may have occurred in the sample while the system was reaching the correct temperature. After this "priming" step, it is important for the pressure due to the sample flow to dissipate completely before the system takes a baseline for the actual measurement. The default fall time for the pressure to reach zero is 5 seconds. However, highly viscous samples will require significantly longer fall times to get accurate measurements. This control is only available when a temperature is entered for the measurement segment.
- **Clear:** Clicking this button will clear all the values from the measurement input table.
- **Syringe Contents Description:** This is a visual indicator of the amount of sample left in the syringe. The fill bar will turn yellow when there is less than 10% of the original sample volume left and red when there is less than 5% left.

Run Parameters

- **Min Rate 1/s:** This is the minimum rate that should be used with the installed VROC Cell and the entered value for the estimated viscosity in order to get good data.
- **Max Rate, 1/s:** This is the maximum rate that should be used with the installed VROC Cell and the entered value for the estimated viscosity in order to get good data. Going over this rate could over-pressurize the VROC Cell and cause permanent damage.
- **Total Measmt Time, s**: This is the total measurement time of all the entered segments, not including the stabilization time (if applicable) and the time necessary for temperature ramp.
- **Total Measmt Vol, ul:** This is the volume required to complete all the measurement segments. The background will be green when this value is 95% of the loaded sample volume or less, yellow when it is between 95% and 100%, and red when it is over 100%.
- **Suggested Syringe Size:** This is the suggested syringe size based on the VROC Cell in use and the estimated viscosity.

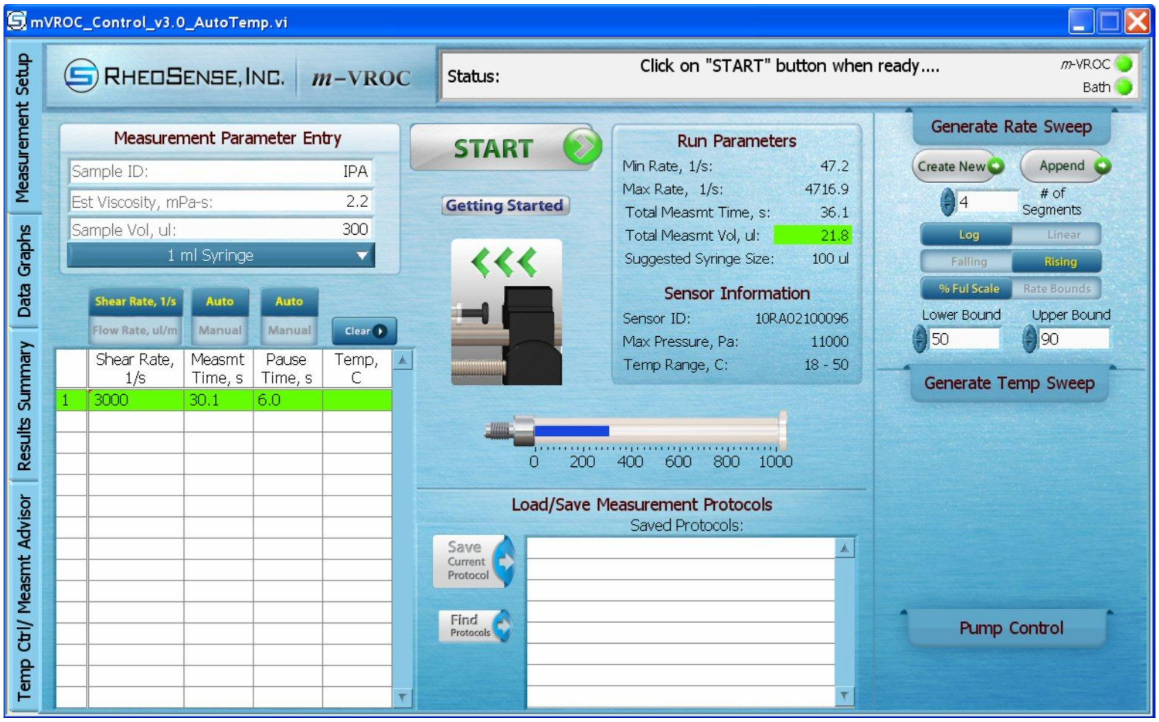
VROC Cell Information

- **Cell ID:** This is the identification or serial number of the VROC Cell, assigned by Rheosense. This information is also printed on the top of the VROC Cell.

- **Max Pressure, Pa:** This is the maximum full scale pressure the VROC Cell has been calibrated to handle. Operating above this pressure could permanently damage the VROC Cell.
- **Temp Range, C:** The allowable operating range of the VROC Cell in degrees Celsius.

Generate Rate Sweep

The rate sweep options appear when the cursor is placed over the "Generate Rate Sweep" area:



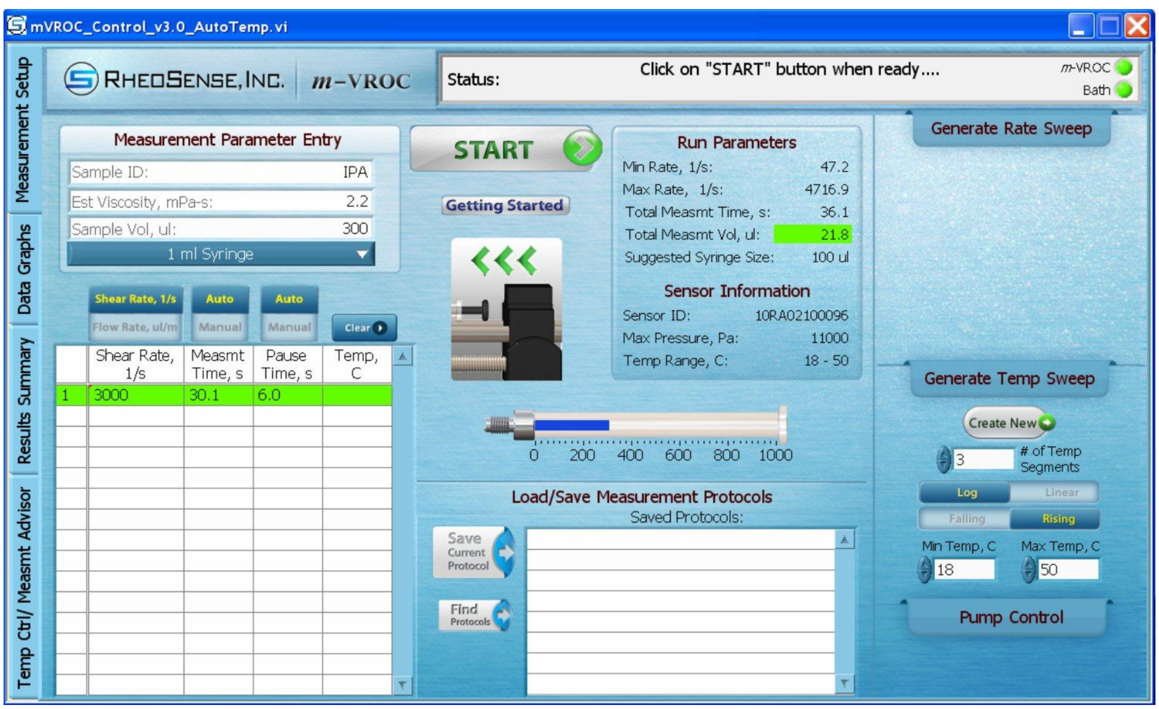
- **Create New:** The 'Generate Rate Sweep' function will generate a number of measurement segments with values determined automatically by the listed parameters. Click on the "Create New" button to automatically populate the measurement table.
- **Append:** Clicking on this button will add additional rate segments in the measurement table rather than replacing what's there. Use this feature to build a measurement run with either increasing or decreasing shear/flow rates (or any combination of increasing and decreasing rates).
- **# of Segments:** This is the number of different rate levels to be run in the measurement.
- **Log/Linear toggle:** This setting determines the spacing of the rate levels when creating a rate sweep. Rate values will be separated by equal values when set this is set to "Linear". When it is set to "Log", rate values will be spaced so that when plotted on a Log scale graph, the distance between points will be equal.
- **Falling/Rising toggle:** This setting determines whether to start the sweep at the slowest rate and then increase (rising), or at the fastest rate and then decrease (falling).
- **% Full Scale/Rate Bounds toggle:** This setting determines on what to base the fastest and slowest segments of the sweep (the end segments). When set to "% Full Scale", the program calculates the end segments based on a percentage of the full scale pressure of the VROC Cell in use. When set to "Manual", the end segments are the

manually entered rates in the numerical controls. The target rates generated are based on the numerical controls for lower and upper bounds, and also the estimated viscosity value entered. Note that if the estimated viscosity is lower than the actual viscosity of the sample, and a rate sweep is generated with an upper bound close to the full scale pressure limit, the run may stop prematurely due to VROC Cell over-pressurization.

- **Lower Bound:** The minimum percentage of full scale pressure the VROC Cell will reach, based on the estimated viscosity entered. This parameter will only be shown when the % Full Scale/Rate Bounds toggle is set to "% Full Scale".
- **Upper Bound:** The maximum percentage of full scale pressure the VROC Cell will reach, based on the Estimated Viscosity entered. This parameter will only be shown when the % Full Scale/Rate Bounds toggle is set to "% Full Scale".
- **Min Rate:** The minimum rate (shear or flow) that the system will measure at. This parameter will only be shown when the % Full Scale/Rate Bounds toggle is set to "Rate Bounds".
- **Max Rate:** The maximum rate (shear or flow) that the system will measure at. This parameter will only be shown when the % Full Scale/Rate Bounds toggle is set to "Rate Bounds".

Generate Temperature Sweep

Note that this feature may only be used with a Thermocube water bath circulator. The temperature sweep options appear when the cursor is placed over the "Generate Temp Sweep" area:



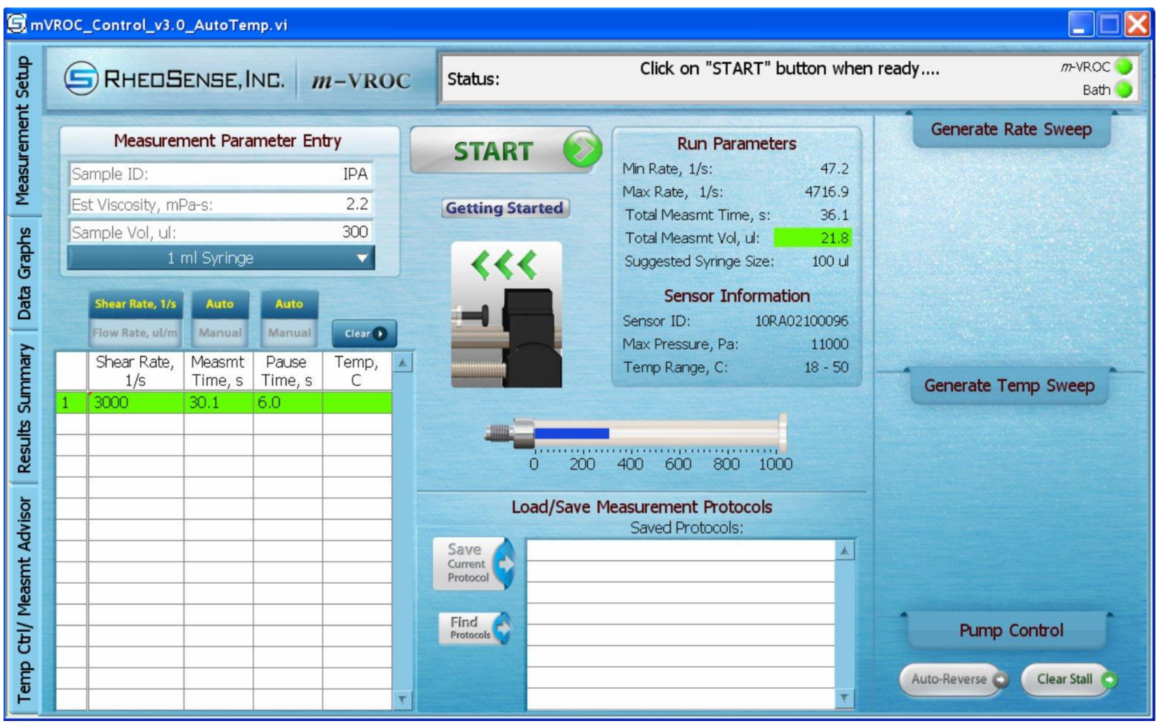
- **Create New:** The 'Generate Temperature Sweep' function will repeat the already listed measurement segments at a number of temperatures determined automatically by the listed parameters. Click on the "Create" button to automatically populate the measurement table.
- **# of Temp Segments:** This is the number of different temperature levels that will be used in a measurement. Keep in mind that each rate segment already entered will

be run at each temperature level, so if there are 3 rate segments entered and a temperature sweep with 3 segments is created, the measurement table will be populated with a total of 9 segments.

- **Log/Linear toggle:** This setting determines how to space the temperature levels in the sweep in the same way the log/linear toggle does in the 'Generate Rate Sweep' function.
- **Falling/Rising toggle:** This setting determines whether to start at a low temperature and then increase (rising), or at a high temperature and then decrease (falling).
- **Min Temp, C:** This is the minimum temperature in the sweep. If this value is outside of the range of the VROC Cell calibration, the measurement table will be populated with the closest in-range value.
- **Max Temp, C:** This is the maximum temperature in the sweep. If this value is outside of the range of the VROC Cell calibration, the measurement table will be populated with the closest in-range value.

Pump Control

The pump control options appear when the cursor is placed over the "Pump Control" area:



- **Auto Reverse:** If the "Auto-Reverse" button is on, the pump pusher block will return to its starting location after the measurement has finished. This feature was designed to be used when there are multiple pre-loaded syringes all with the same volume that need to be measured. In that case each successive syringe can be loaded and run without releasing the pusher block from the lead screw.
- **Clear Stall:** If the pusher block ever becomes jammed, click on this button.

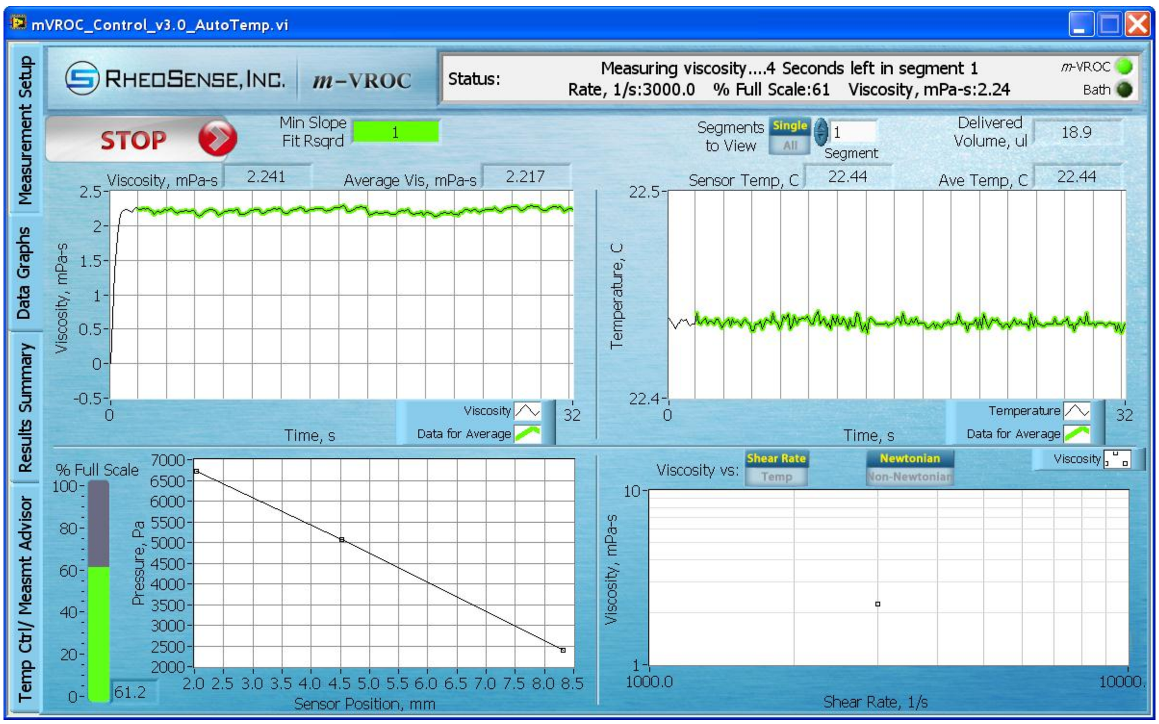
Load/Save Measurement Protocols

Saved protocols are displayed in this area for easy access.

- **Save Current Protocol:** Click on this button to save the entered values into a protocol. Give a name to the protocol when the dialog appears. Protocols are saved in the '\....\(My) Documents\LabVIEW Data\Measurement Protocols' folder by default.
- **Find Protocols:** Click on this button to navigate to a folder that contains saved protocols.

ii. Data Graphs Tab

The front panel (user interface) looks like this:



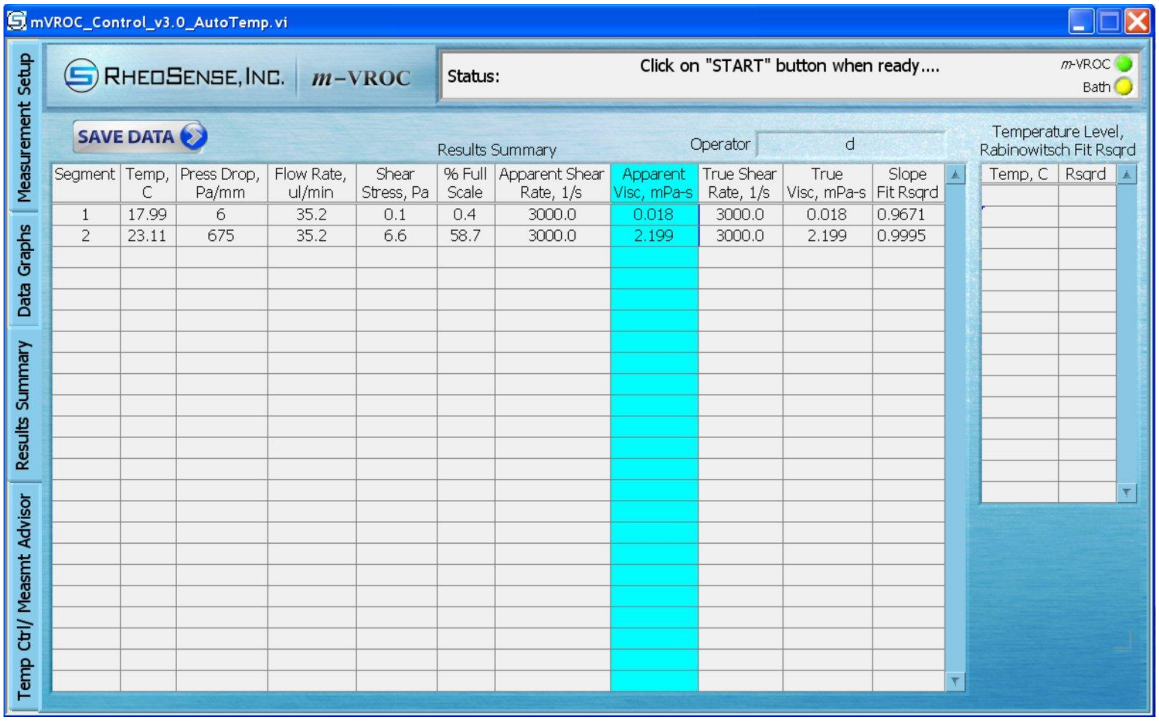
- **Stop:** Clicking on this button halts a measurement at any point after clicking "Start". This button only appears during a measurement in the upper left corner.
- **Save Data:** After a measurement is complete (or the "Stop" button is clicked), this button will be available in place of the "Stop" button. Click on this button to save another copy of the data. See section 6c for details.
- **Min Slope Fit Rsqrd:** This is an indicator of how close the VROC Cell pressure readings are to a straight line. Ideally this number should be 1, indicating that the pressure readings form a perfectly straight line. If the value is below 0.98, the background will turn red. This could be an indication that the viscosity reading has not yet reached steady state, or that something has occurred to compromise the reading. This value represents the minimum value or "worst" data point for all the data points used for the average value. This value will often be below 0.98 until enough data for the average value is collected.
- **Include/Exclude Rsqrd toggle:** This setting allows measurement segments with "Ave Slope Fit Rsqrd" values less than 0.98 to be excluded from the displayed data. This is useful for high segment numbers or long measurements where a bubble or

one bad data point may ruin the whole set. This toggle only appears after a measurement completes and the original data is saved. The data set may then be re-saved excluding the bad points.

- **Single/All Segments to View toggle:** This setting switches the plots between displaying all segments at once or individual segments. When segments are displayed individually, the "Viscosity vs. Time" and "Temperature vs. Time" plots show the data used to calculate the average value highlighted in green. Additionally, the average values for viscosity and temperature are shown along with the instantaneous values. Use the numerical control to cycle through the displayed segments.
- **Segment:** When "Segments to View" is set to "Single", use this control to display the different measurement segments in the graphs.
- **Delivered Volume, ul:** This is the total volume delivered for the current measurement run.
- **Viscosity, mPa-s vs. Time, s graph:** This shows the value of the viscosity as the measurement proceeds.
- **Viscosity, mPa-s:** This is the instantaneous viscosity reading of the current measurement segment.
- **Average Vis, mPa-s:** This is the average viscosity reading of a measurement segment when the "Segments to View" button is set to "Single". NaN means not a number, and is displayed until sample is being pumped through the VROC Cell.
- **VROC Cell Temp, C:** This is the instantaneous temperature reading of the current measurement segment, shown in real time.
- **Ave Temp, C:** This is the average temperature reading of a measurement segment when the "Segments to View" button is set to "Single".
- **Temperature, C vs. Time, s graph:** This shows the temperature of the VROC Cell as the measurement proceeds.
- **% Full Scale Pressure bar:** This shows the percentage of full-scale pressure being detected by the VROC Cell. If "Segments to View" is set to "All", this indicator shows the maximum pressure reading from all segments. If "Segments to View" is set to "Single", this indicator shows the instantaneous reading of a currently running segment or the last value of a completed segment. The fill bar of this indicator will turn red if the value goes above 100%, or yellow if the value is below 5%.
- **Pressure, Pa vs. Sensor Position, mm graph**: This shows the plot(s) of pressure vs. sensor position within the VROC Cell flow channel. Instantaneous values are shown for the currently running segment, while average values are shown for completed segments. Each plot should be a straight line. See the description of "Min Slope Fit Rsqrd" for more information.
- **Viscosity vs. Shear Rate/Temp toggle:** This switches the x-axis of the viscosity plot between shear rate and temperature.
- **Newtonian/Non-Newtonian toggle:** If the fluid being measured is Non-Newtonian (the viscosity changes with shear or flow rate), click this button to apply the Weissenberg-Rabinowitsch-Mooney correction and obtain a value for the "true" viscosity. The corrected values will be shown in red in the plot along with the uncorrected values in black. Note that there must be at least 3 rate segments to get a meaningful Rabinowitsch correction.
- **Viscosity, mPa-s vs. Shear or Flow Rate:** This shows the instantaneous measured viscosity as the shear or flow rate changes.

iii. Results Summary Tab

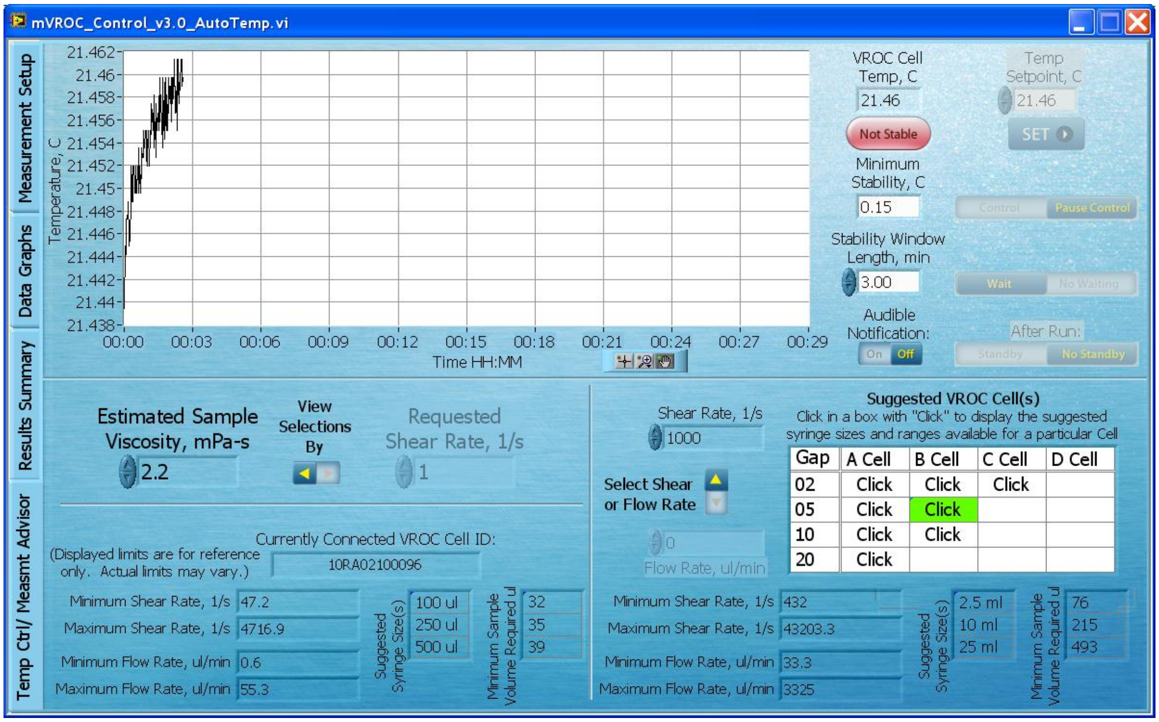
The results summary table lists the average values for each measurement segment. The front panel (user interface) looks like this:



- **Stop:** Clicking on this button halts a measurement at any point after clicking “Start”. This button only appears during a measurement in the upper left corner.
- **Save Data:** After a measurement (or the “Stop” button is clicked) this button will be available in place of the “Stop” button. Click on this button to save another copy of the data. See section 6c for details.
- **Temperature Level, Rabinowitsch Fit Rsqrd table:** If the Newtonian/Non-Newtonian toggle button on the Data Graphs tab is set to “Non-Newtonian”, this table will show the R^2 correlation value of the correction for each temperature level. If the R^2 value is less than 0.98 for a temperature level, the correction may not be very accurate. Note that there must be at least 3 shear or flow rate segments to get a meaningful Rabinowitsch correction.

iv. Temperature Control/Measurement Advisor Tab

This tab is for use with temperature control, and/or to help in determining appropriate VROC Cell type, syringe size, and measurement requirements. The front panel (user interface) looks like this:



- **Temperature, C vs. Time HH:MM:** This shows the temperature of the VROC Cell while the instrument is on and the software is running. Only the most recent 30 minutes of data is displayed. When the Thermocube water bath is connected and running, the plot of the water bath's temperature will be displayed in red. A slight temperature difference between the VROC Cell and the water bath is normal.
- **VROC Cell Temp, C:** This is the current temperature of the VROC Cell.
- **Stable/Not Stable indicator:** This indicates whether the temperature is stable, as defined by the "Minimum Stability, C" and "Stability Window Length, min" control settings.
- **Temp Setpoint, C:** This is the desired operating temperature selected by an operator. Note that this button only becomes active when a Thermocube water bath circulator is in use.
- **Set:** After entering a temperature set point, click on this button to accept the new value. Note that this button only becomes active when a Thermocube water bath circulator is in use.
- **Minimum Stability, C:** This defines the minimum stability condition to indicate whether the temperature has reached equilibrium. The peak to peak temperature variation must be within this value centered around the temperature set point for the specified amount of time. For example, for the default value of 0.15 °C, the temperature cannot be greater than +0.075 °C over the set point, or -0.075 °C below the set point.
- **Control/Pause toggle:** This starts or pauses temperature control. Note that this only becomes active when a Thermocube water bath circulator is in use. When changing or adding more sample to the system while under temperature control, there will be a disturbance. To prevent the Thermocube controller from trying to compensate during this time, set this toggle to "Pause Control" and the Thermocube controller will continue to operate at its current value until the toggle is set back to "Control". This will minimize the time it takes the temperature to reach equilibrium after a new sample is loaded.

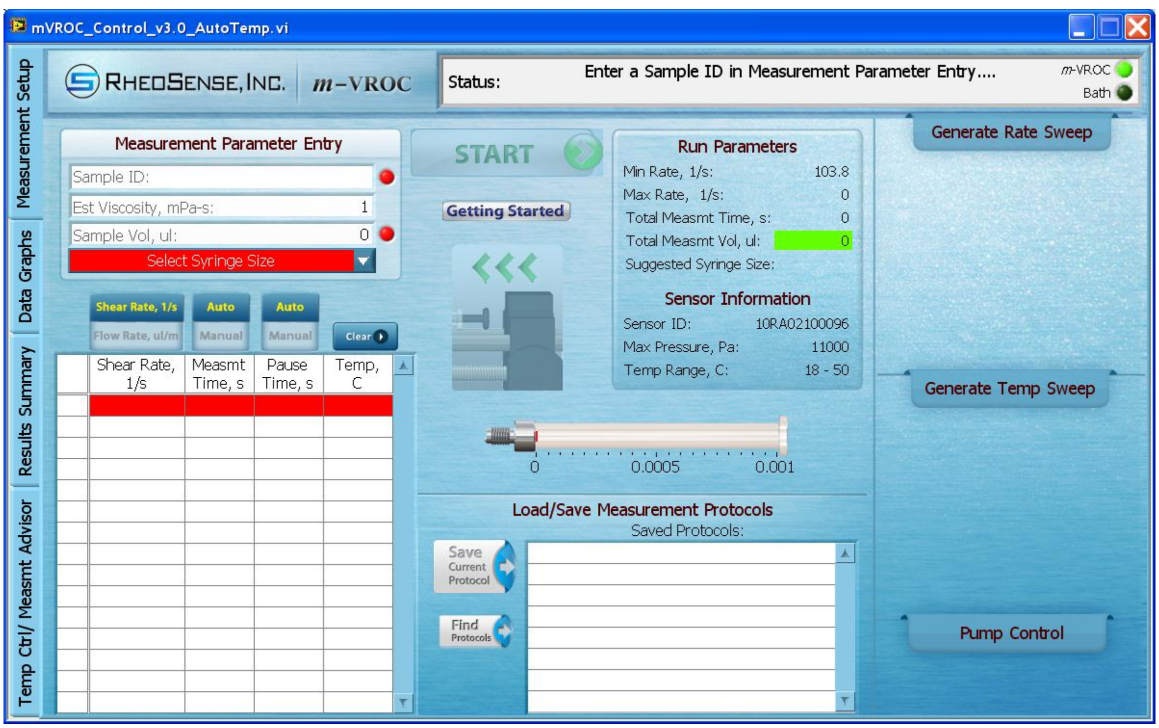
- **Stability Window Length, min:** This is the length of time during which the temperature must meet the minimum stability requirement.
- **Wait/No Waiting toggle:** When this control is set to “Wait”, the program will wait for the temperature to stabilize before running a measurement. Note that this only becomes active when a Thermocube water bath circulator is in use.
- **Audible Notification On/Off toggle:** When this control is set to “On” the program will beep when the temperature becomes stable. The beep alerts the user when the system is ready for a manual measurement at the desired temperature.
- **After Run Standby/No Standby toggle:** When this control is set to “Standby” the water bath will be set to standby mode after a run. Note that this only becomes active when a Thermocube water bath circulator is in use.
- **Estimated Sample Viscosity, mPa-s:** Enter the estimated viscosity of the sample. The range of the VROC Cell will be displayed with units of shear rate (1/s).
- **View Selections By toggle:** This switches the displayed measurement range of the VROC Cell between shear rate and viscosity.
- **Requested Shear Rate, 1/s:** Enter the shear rate at which the measurement will be taken. The range of the VROC Cell will be displayed with units of viscosity (mPa-s).
- **Currently Connected VROC Cell ID:** The ID of the VROC Cell in use is displayed in this field.
- **Minimum Viscosity, mPa-s:** This is the minimum viscosity required to get an accurate reading at the entered value for shear rate. This value will be displayed when the “View Selections By” toggle is set to “Requested Shear Rate, 1/s”.
- **Maximum Viscosity, mPa-s:** This is the maximum viscosity measurable at the entered value for shear rate. This value will be displayed when the “View Selections By” toggle is set to “Requested Shear Rate, 1/s”.
- **Minimum Shear Rate, 1/s:** This is the minimum shear rate required to get an accurate reading at the entered value for viscosity. This value will be displayed when the “View Selections By” toggle is set to “Estimated Sample Viscosity, mPa-s”.
- **Maximum Shear Rate, 1/s:** This is the maximum shear rate achievable at the entered value for viscosity. This value will be displayed when the “View Selections By” toggle is set to “Estimated Sample Viscosity, mPa-s”.
- **Minimum Flow Rate, ul/min:** This is the minimum flow rate required to get an accurate reading for either the entered viscosity or shear rate value.
- **Maximum Flow Rate, ul/min:** This is the maximum flow rate achievable at either the entered viscosity or shear rate value.
- **Suggested Syringe Size(s):** This (these) is (are) the smallest syringe size(s) that will allow the system to measure viscosity within the displayed ranges. Syringes larger than those displayed may work, but will most likely require a larger sample volume.
- **Minimum Volume Required, ul:** This is the smallest amount of sample volume that is required in order to take a measurement at a single shear or flow rate.
- **Suggested VROC Cell(s):** To use this table, enter the desired parameter(s) in the field(s) to the left of the table. Click on any box in the table with the work “Click” in it to display the capabilities of that particular VROC Cell.
- **Shear Rate, 1/s**: Enter a desired shear rate in this field. This control is only available when the “View Selections By” toggle is set to “Estimated Sample Viscosity, mPa-s”.
- **Select Shear or Flow Rate toggle:** Click on this to switch between being able to use a shear rate or a flow rate to determine VROC Cell needs. This control is only available when the “View Selections By” toggle is set to “Estimated Sample Viscosity, mPa-s”.

- **Flow Rate, ul/min:** Enter a desired flow rate. This control is only available when the "View Selections By" toggle is set to "Estimated Sample Viscosity, mPa-s".

B. BASIC SOFTWARE OPERATION

After the software has been installed and the computer re-started, power on the *m*-VROC unit with a VROC Cell connected. Plug one end of the USB cable into the *m*-VROC unit and the other end into a free USB port on the computer. The computer will indicate that it has found new hardware and is installing a driver. Start the *m*-VROC control program using either the shortcut that was placed on the desktop during installation or from the computer's Start Menu folder called '*m*-VROC Control'. When the program opens, it will attempt to connect to the *m*-VROC unit. The status bar in the upper right corner will display "Initializing Instrument…"

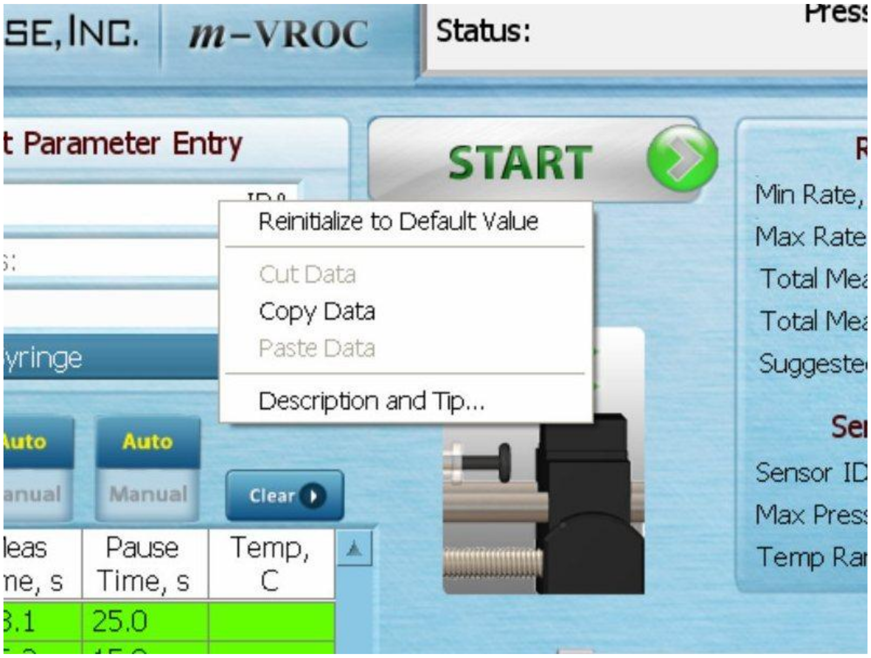
After communication has been established, several of the fields in the "Measurement Parameter Entry" area will have red backgrounds or blinking lights next to them. This indicates that either there is no value entered or that the entered value is not acceptable. During this time, it will not be possible to start a measurement, and the "Start" button will appear dim. The status bar will display what needs to be entered or changed. For instance, the first message displayed on initial startup is, "Enter a Sample ID in Measurement Parameter Entry….":



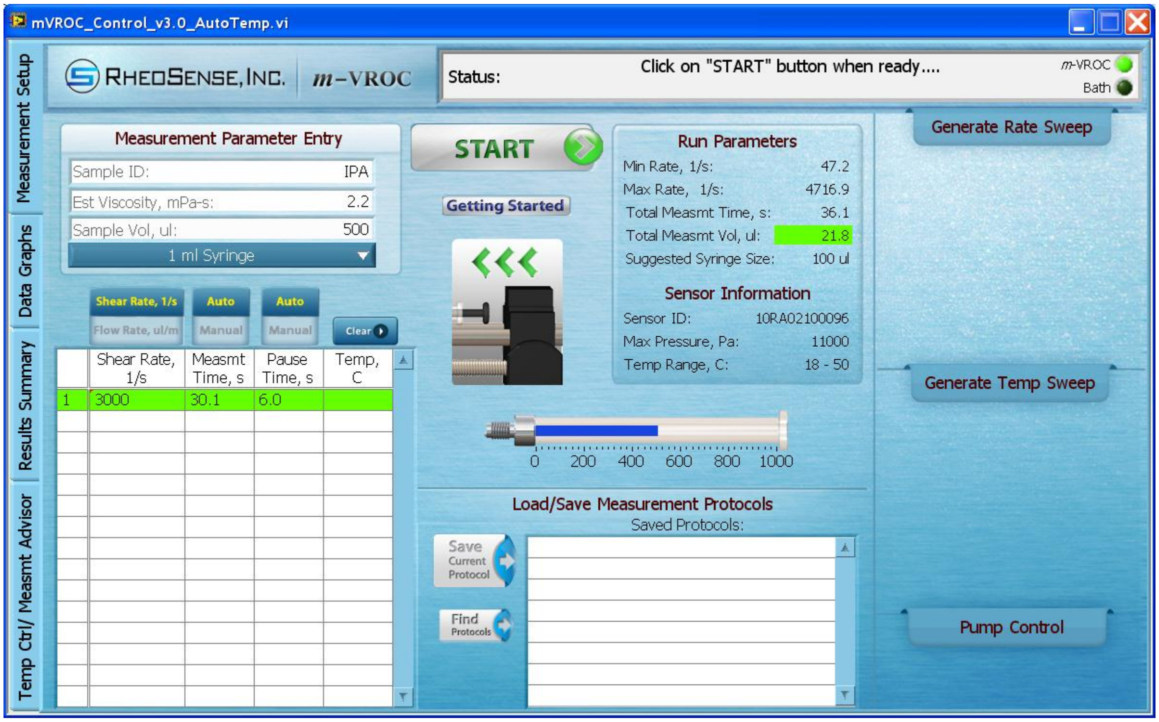
Once an acceptable value has been entered, the red background or blinking light for that parameter will disappear. After the first time the program is started and run, most of the settings will be saved to a configuration file that will reload those settings on subsequent program starts. There are two areas where settings will not be reloaded: the "Sample Vol, ul" field and the measurement input table. The sample volume should always be confirmed when starting the program at the beginning of a data collection run, especially if a partially full syringe was left in the instrument on shutdown. To re-use measurement parameters entered in the measurement input table, use the

Load/Save Measurement Protocols feature.

Most fields and controls in the software can be right-clicked for more information. Right-clicking will bring up a menu with a "Description and Tip" option. Clicking on this option will bring up more information about that feature:



- To perform a measurement at a single shear or flow rate:
1. Start the *m*-VROC control software.
2. Load a syringe with sample into the instrument (see section 5).
3. Enter a sample ID.
4. Enter the estimated viscosity.
5. Enter the volume of sample in the syringe. If the exact amount is uncertain, underestimate the sample volume.
6. Select the syringe size from the pull-down menu.
7. Select the rate type, either shear or flow.
8. Enter a rate (with a value between the minimum and maximum values listed under "Run Parameters") into the left column of the table.
9. Leave the "Measmt Time" and "Pause Time" columns blank and both set to "Auto". A value will be entered automatically.
10. At this point, both the "Start" button and the 'move pusher block to plunger' icon will become bright, indicating they are available. The status bar will display, "Click on "START" button when ready.... ":



11. Click on the 'move pusher block to plunger' icon to remove the gap left between the pusher block and the syringe plunger when the syringe was loaded.
12. Click on the "Start" button to start the measurement. The program will automatically bring up the Data Graphs tab to display the results as the measurement progresses.

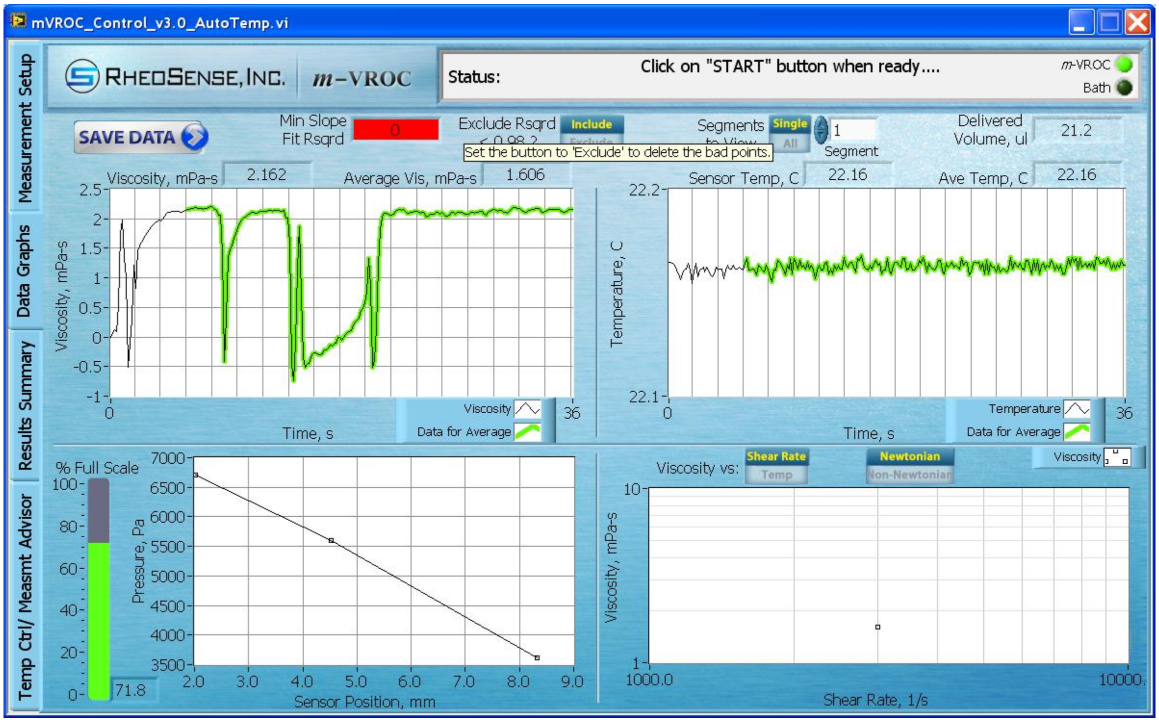
Once the measurement has begun, it is possible to switch between the tabs. A "Stop" button will appear in the upper left corner in the Data Graphs and Results Summary tabs, as well as in the Measurement Setup tab where the "Start" button was. Clicking on the "Stop" button in any tab will halt the measurement.

After the measurement completes (or after the "Stop" button has been clicked), the data will automatically be saved. See section 6c for more information on saving data. The measured viscosity value will be displayed in the Data Graphs tab in the field labeled "Average Vis, mPa-s", as well as in the Results Summary tab in the blue highlighted column.

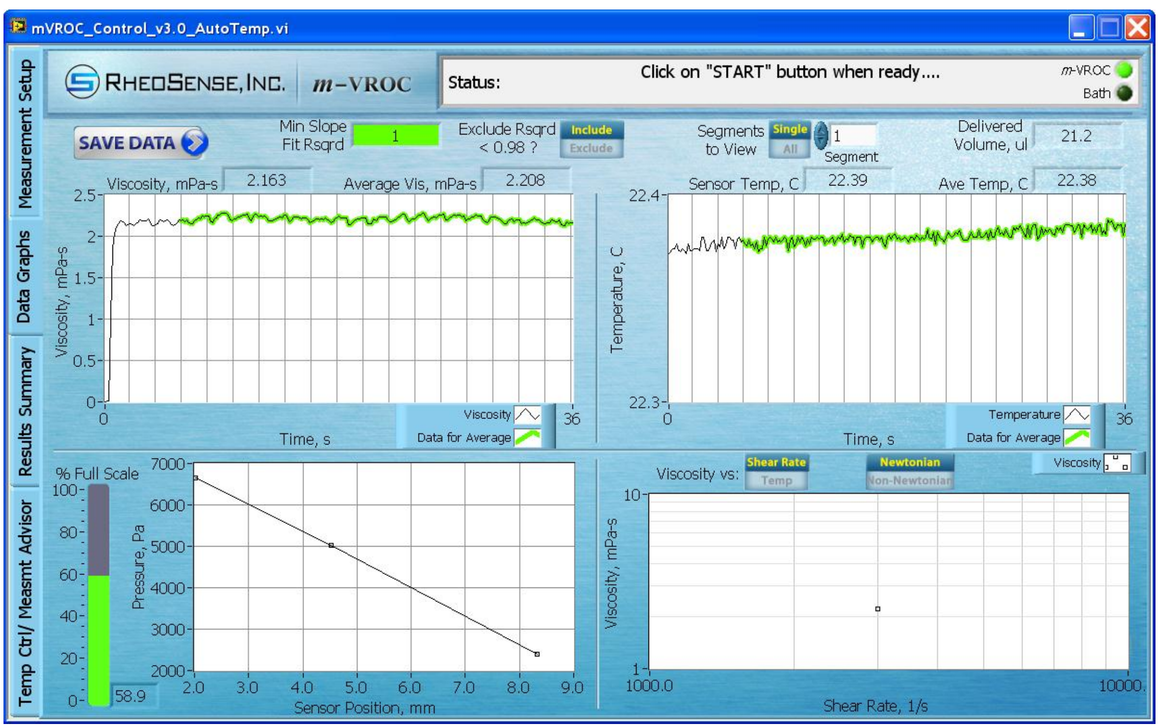
The basic indicators that the measurement result is accurate are:

- The background of "Min Slope Fit Rsqrd" should be green, indicating the minimum value is above 0.98.
- The plot of "Viscosity vs. Time" in the upper left corner in the Data Graphs tab should be a level, continuous line (minor oscillations are ok) for the highlighted green portion.
- The "% Full Scale" indicator in the lower left corner in the Data Graphs tab should be green.

A measurement with an INACCURATE result might look like this:



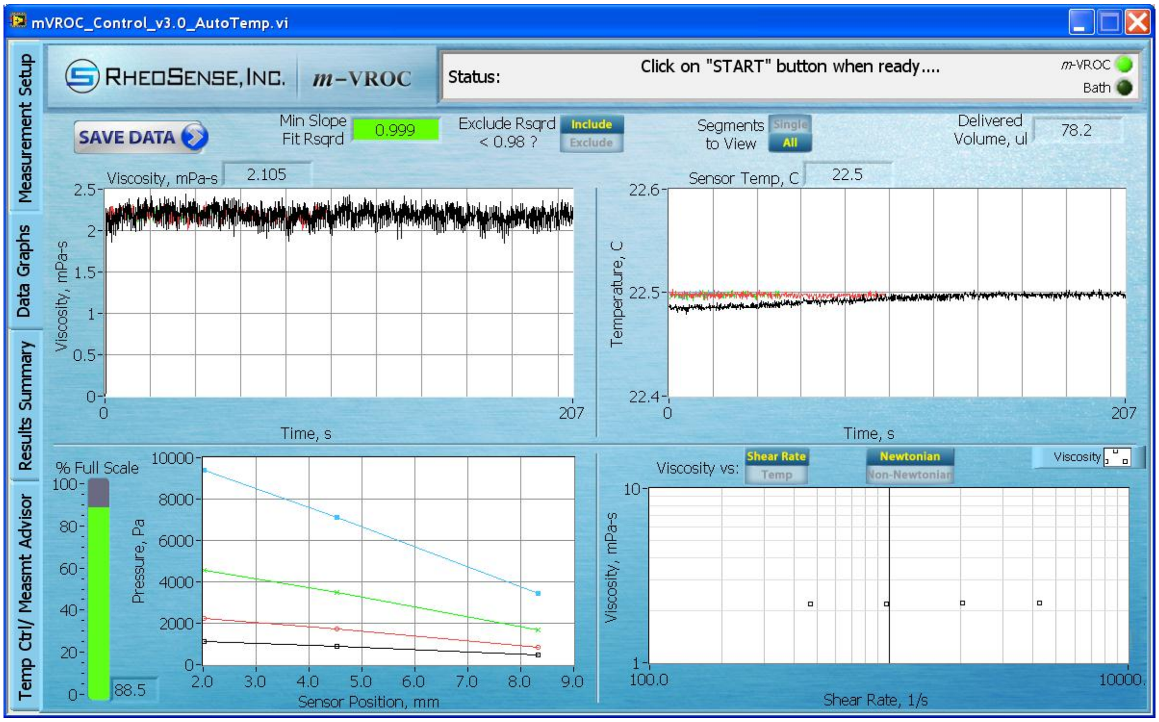
A measurement with an ACCURATE result might look like this:



- To perform a measurement at multiple shear or flow rates:
  1. Follow steps 1-6, above.
  2. Enter multiple rates in the table manually, or automatically populate the table using the 'Generate Rate Sweep' function.

3. Leave the "Measmt Time" and "Pause Time" columns blank and both set to "Auto". A value will be entered automatically.
4. Click on the "Start" button.

A multi-rate measurement with an accurate result might look like this:



- To perform a measurement at one or more temperatures (with the optional Thermocube water bath connected):
  1. Follow the instructions on setting up the temperature control hardware in section 4b.
  2. Enter the desired rate(s) in the table.
  3. Enter a temperature for each rate in the temperature column. Only temperatures within the temperature range of the VROC Cell as shown under "VROC Cell Information" are allowed. Alternatively, use the 'Generate Temperature Sweep' function to populate the temperature values. This function will repeat each of the rate segments in the table for each temperature segment.
  4. Click on the "Start" button.
  5. Once the temperature stabilizes (based on the stabilization parameters entered), the measurement will begin.

A multi-rate, multi-temperature measurement might look like this:

C. Saving Data

After a measurement or after the “Stop” button is clicked, data is saved automatically to the ‘\....\(My) Documents\LabVIEW Data\Test Logs’ folder. This folder is created when the program is installed. Two different records are created for each test. One is an Excel formatted spreadsheet file that contains a test summary, the test parameters, and all of the raw data. The other is a PDF file that contains a test summary, the test parameters and 3 plots (Viscosity vs. Time, Viscosity vs. Shear Rate (or Temperature), and Pressure vs. Position). These automatically saved files have names composed of the Sample ID and the time stamp when the test ended, with either a .xls or .pdf file extension (e.g., IPA_1_5_2012 2h03m47sPM.pdf). Note that the computer used to run the *m*-VROC instrument must have Microsoft Excel installed in order for the program to create an Excel-formatted data file.

In the Data Graphs tab there are four toggle switches that either modify the data or change the way it is displayed in the graphs: “Include/Exclude” (Rsqrd), “Single/All”, “Shear Rate/Temp”, and “Newtonian/Non-Newtonian”. The settings of these switches modify the way the data is saved to the two log files. When the data is saved automatically, only the “Shear Rate/Temp” and “Newtonian/Non-Newtonian” toggle are active. All the data is always included in the automatically saved Excel data file, and the plot in the automatically saved PDF file will show all the measurement segments. The “Viscosity vs. Shear Rate/Temp” graph will reflect the setting of the toggle, and the Weissenberg-Rabinowitsch-Mooney correction will be applied to the data if the toggle is set to “Non-Newtonian”.

After the data is saved automatically, it may be desirable to save another copy of the data with different settings of the toggle switches, or to give the data files different names. For instance, if a measurement with many segments was performed and a small bubble caused a momentary dip in the viscosity reading, setting the "Include/Exclude" (Rsqrd) toggle to "Exclude" may remove that bad data point from the segment to give a more accurate result, making it unnecessary to repeat the measurement. After the data is automatically saved, the status bar will display: "Click on "Start" button when ready…". It is then possible to change the settings of the toggle switches as desired, and then click on "Save Data". The data may be saved manually as many times as desired, and will remain available until the "Start" button is clicked again.

D. Determining Measurement Time, Pause Time, and Fall Time

In most cases the "Auto" setting for the measurement and pause times, and the default value (5 seconds) for the fall time will be sufficient to get accurate results. However, there are instances where using the manual setting and selecting specific values will yield better results:

- **The measurement time under the "Auto" setting is 1 second:** When the "Auto/Manual" (measurement time) toggle is set to "Auto" the program calculates the amount of time necessary for the pump lead screw to complete one rotation for the given shear or flow rate. Sometimes this time will be 1 second. With the pause time also set to "Auto" (it will be 3 seconds in these instances), only 25% of the sample used for that measurement segment will go toward calculating the viscosity. Depending on the rise time of the sample for such measurements, it may be possible to decrease the pause time to less than 3 seconds to conserve sample. Take a single segment measurement with the "Auto" setting selected for both the measurement time and pause time, and observe the rise time (the amount of time it takes for the sample to reach a steady state viscosity value) in the upper left graph in the Data Graphs tab. For subsequent measurements, if the observed rise time is less than 3 seconds set the pause time to "Manual" and enter the observed rise time plus 20%. If sample is plentiful, consider subtracting a second from the pause time and adding it to the measurement time.
- **High viscosity samples are being measured:** Highly viscous samples may have rise times longer than the pause times selected by the program in "Auto" mode, especially if any bubbles become trapped during sample loading. When dealing with highly viscous samples it is best to first do a test or "dummy" run to find out what the rise time of the sample is for a particular shear or flow rate. In general, rise times increase as shear/flow rates decrease. If a multiple rate measurement is to be done, determine the rise time of the sample at the lowest shear or flow rate first. Then, set the pause time to "Manual", add 10-20% to that time, and use the resulting value as the pause time for all the measurement segments.
- **High viscosity samples are being measured with temperature control:** After the temperature has stabilized, the instrument will push a small amount of sample through the VROC Cell before collecting data. This minimizes the effects from bubbles that may appear in the sample as the system is approaching the correct temperature. After this "priming" step, it is important for the pressure (due to the sample flowing through the VROC Cell) to dissipate completely before the system takes a baseline for the actual measurement. The default fall time for the pressure to reach zero is 5 seconds. However, highly viscous samples or those with large

internal structures (e.g., emulsions) will require significantly longer fall times. To determine the correct fall time for such samples, do a single rate measurement (set "Measmt Time" and "Pause Time" to "Auto"). As soon as the pump stops moving (i.e., the measurement ends), start a timer. After about 30 seconds, click the "Start" button to begin another measurement. When the status bar displays "Measuring Baseline…", stop the timer. Allow the measurement to proceed. When the measurement ends, compare the viscosity value of the first run with that of the second run. If the values are equal, the amount of time recorded on the timer is a sufficient fall time. If the second value is LOWER than the first value by more than a few percent, a longer fall time will be needed. Wait for about a minute after the second measurement completes, then repeat the measurement a third time. Compare the third measurement with the first one. If the results are equal, the time between the second and the third measurement is a sufficient fall time. Repeat this process (increasing the time in increments of 30 seconds each time) until the most recent viscosity value equals that from the first measurement. In some cases, sufficient fall times may be on the order of a couple of minutes.

7. Cleaning and Maintenance

A. Cleaning the VROC Cell After Use

⚠ **Always use the provided software with the *m*-VROC for any testing and cleaning. Do not attempt to manually push cleaning fluid through the VROC Cell.**

Drying of a sample solution inside the VROC Cell after a prolonged period of inactivity carries a high risk of permanently clogging the VROC Cell and causing irreversible damage. To avoid clogging, it is very important to clean the VROC Cell thoroughly with a compatible solvent to remove sample solution after use. The recommended procedure for cleaning after running low to medium viscosity samples is:

1. Unload the syringe filled with the test sample (reverse the steps in section 5b).
2. Wipe and clean the sample solution from the inlet fitting on the VROC Cell assembly.
3. Connect a solvent-filled syringe to the VROC Cell.
4. In the Measurement Setup tab, enter a flow rate (this should not exceed the maximum used during sample testing) that will command the software to push about 250 µl of solvent through the sensor (the number in the "Total Measmt Vol, ul" field will be about 250). This will remove the majority of the sample solution from the VROC Cell.
5. Increase the flow rate (this will be based on the viscosity of the solvent), and flush the VROC Cell two to three times.
6. Stop infusing and let the solvent sit for one to two minutes.
7. Return to a low flow rate and flush the VROC Cell two to three more times.
8. Repeat steps 5 through 7 as many times as necessary (the number of repetitions will depend on the type of sample solution). Please see Appendix II for more on the suggested cleaning procedure.
9. Finally, test the viscosity of the solvent with the VROC Cell to verify that the VROC

Cell is clean.

10. After cleaning the VROC Cell, unload the syringe.
11. Wipe and clean the inlet fitting on the VROC Cell assembly again, and make sure the valve is closed (the small arm will be vertical).

After measuring high viscosity samples, it can be tricky to thoroughly clean the VROC Cell in preparation for measuring lower viscosity fluids or for storage. If a high viscosity sample is not completely removed before running a lower viscosity sample, erroneous measurements may result. In general, it is not necessary to do any special cleaning procedure when switching between samples of similar viscosities and miscibilities.

The recommended procedure for cleaning after running high viscosity samples is:

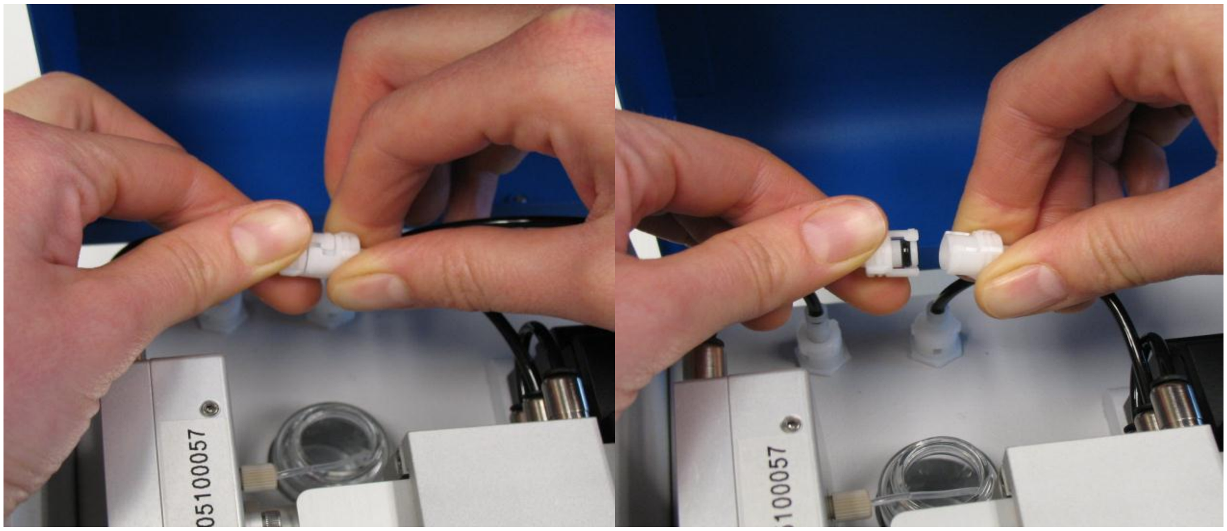
1. Use a low viscosity cleaning fluid.
2. Fill a syringe with the cleaning fluid and mount it in the unit (follow the steps in section 5b).
3. In the Measurement Setup tab, select a flow rate low enough that half the volume of the fluid in the syringe will be run through the sensor in no less than 5 to 10 minutes. It is ok if the software indicates that the selected flow rate is too low to get a good viscosity reading (i.e., the background of the measurement segment row in the table is yellow).
4. After step 3 is complete, raise the flow rate in the Measurement Setup tab to push the rest of the cleaning fluid through the sensor (high enough that the sensor reads about half its full scale pressure if possible).
5. Check to make sure the viscosity reading of the cleaning fluid is accurate. If it is not, repeat steps 1 through 4.
6. Once the reading is correct, proceed to the next sample to be tested.

If clogging occurs or persists, do not attempt to force a flow as this may cause permanent damage to the VROC Cell. If there is any sign of clogging, please contact RheoSense for assistance.

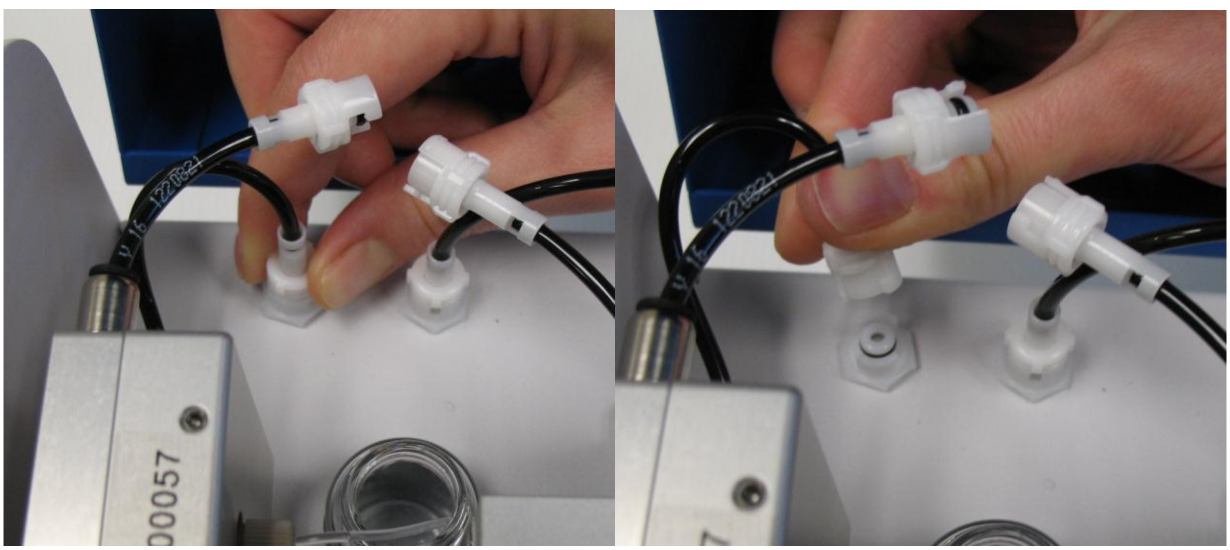
B. Changing the VROC Cell

1. If there is still a syringe connected to the VROC Cell assembly, remove it (reverse the steps in section 5b). Make sure the valve is closed (the small arm will be vertical).
2. Unplug the connector from the bottom of the VROC Cell assembly.

3. Grasp the connector joining the VROC Cell to the syringe jacket, and twist counter-clockwise:

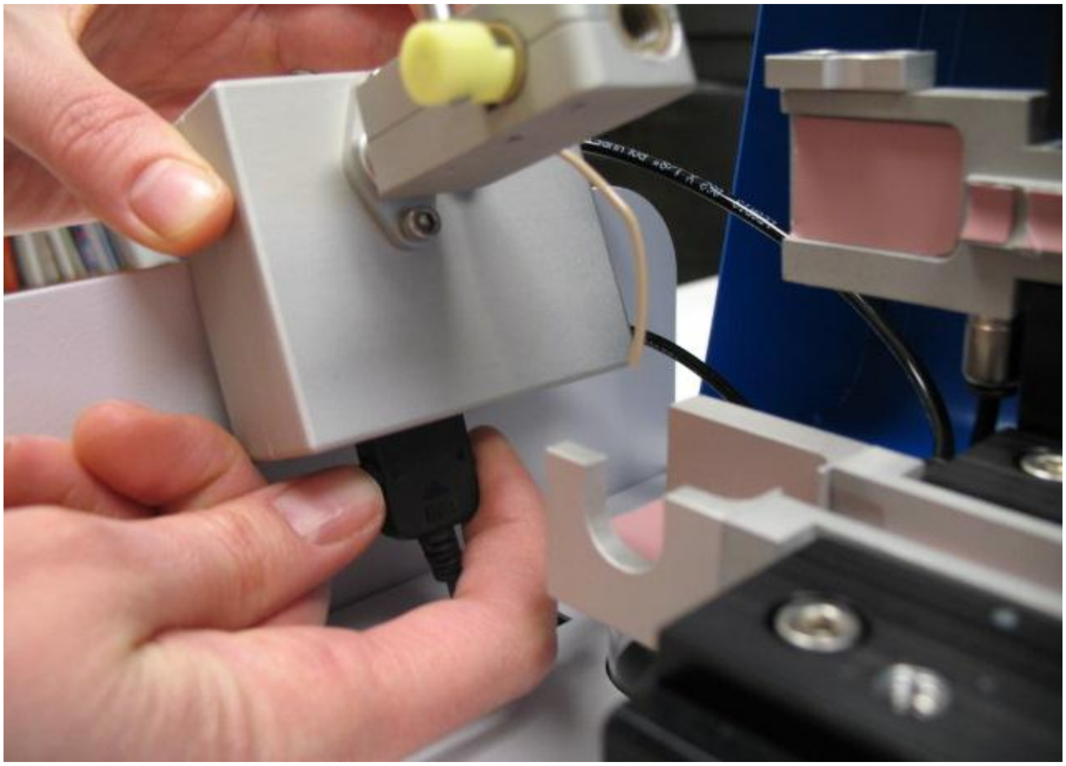


4. Disconnect the VROC Cell assembly from the unit by grasping the connector and twisting counter-clockwise:

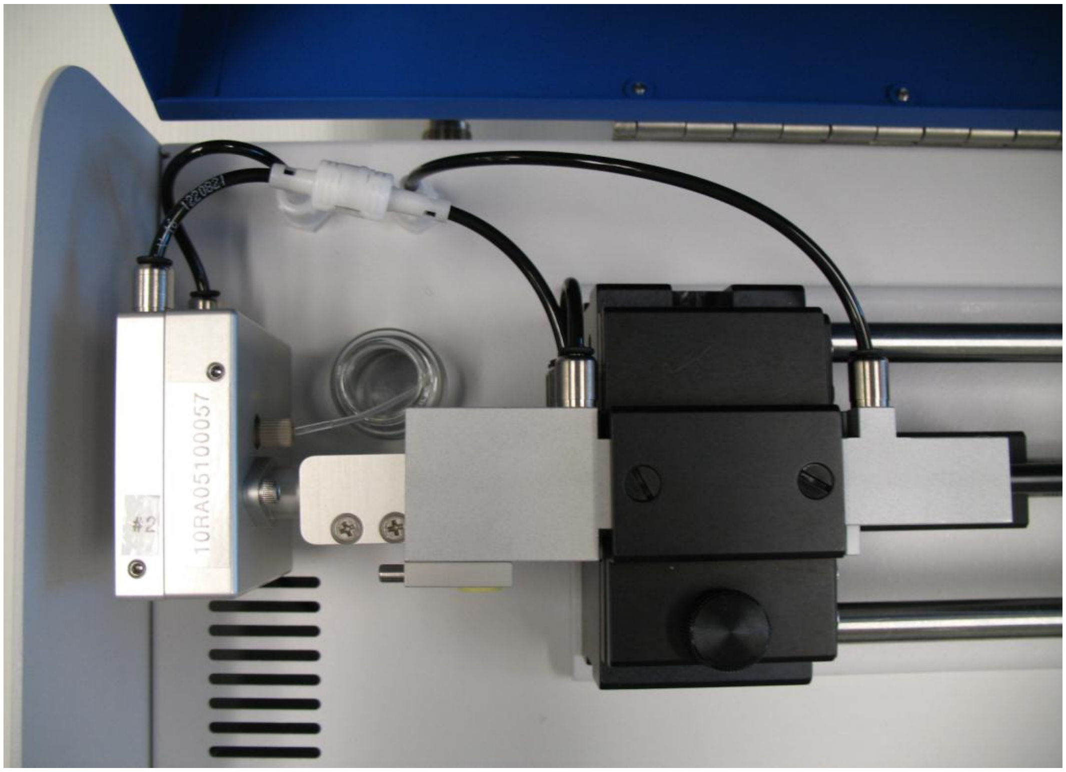


5. Choose the desired VROC Cell assembly. Connect the VROC Cell by twisting the connector clockwise, in the same locations as in steps 3 and 4 above.

6. Connect the bottom of the VROC Cell assembly with the plug from the base of the *m*-VROC unit (Note that the plug only connects one way. If connecting the plug to the assembly is difficult, rotate the plug 180° and try again.):



7. Make sure the tubes are connected correctly, and the waste tube is placed in the waste bottle:

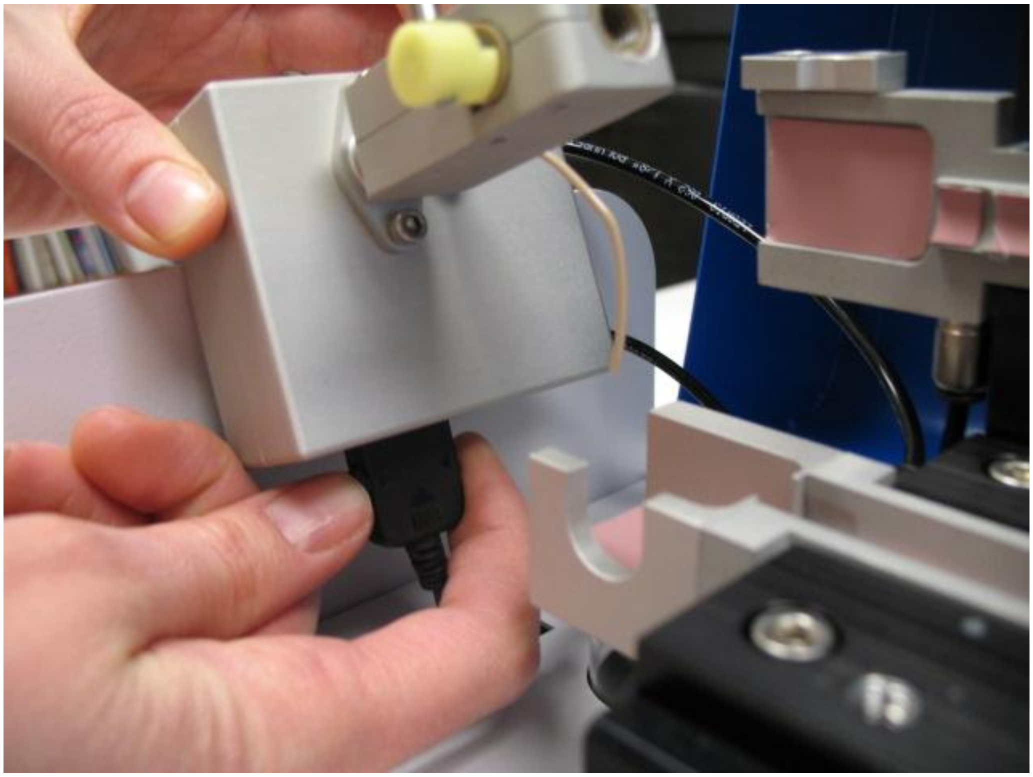


C. Changing the Syringe Jacket

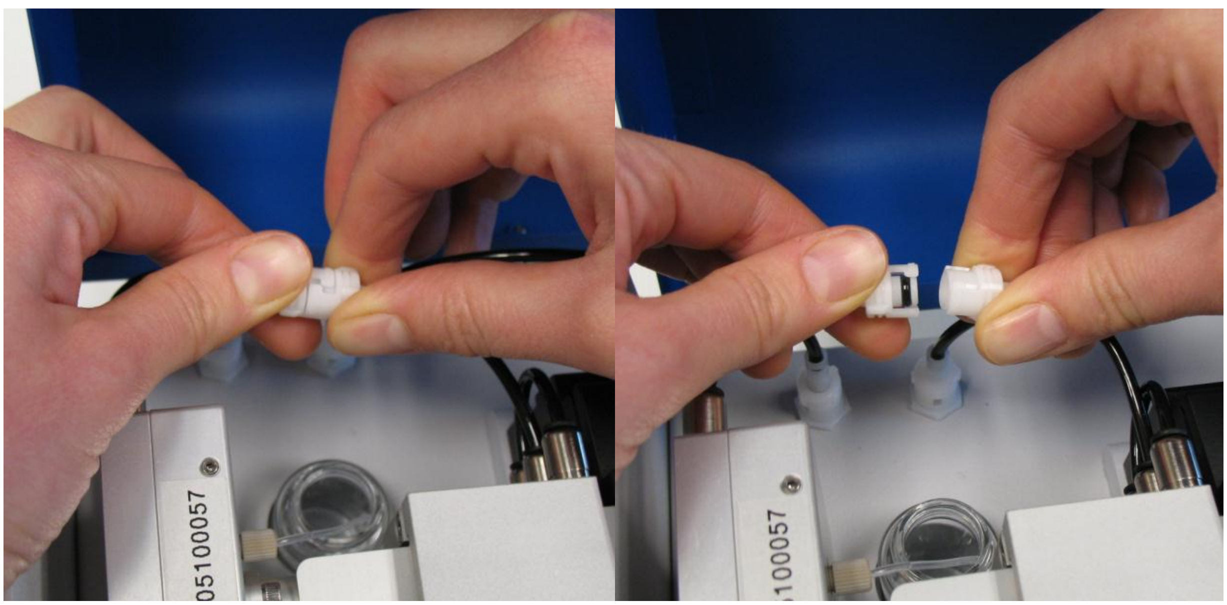
A hardware kit is included for changing the syringe jacket. It contains spare 10-32 screws and a 5/32" Allen wrench:

If the kit is missing any of these components, contact RheoSense for assistance.

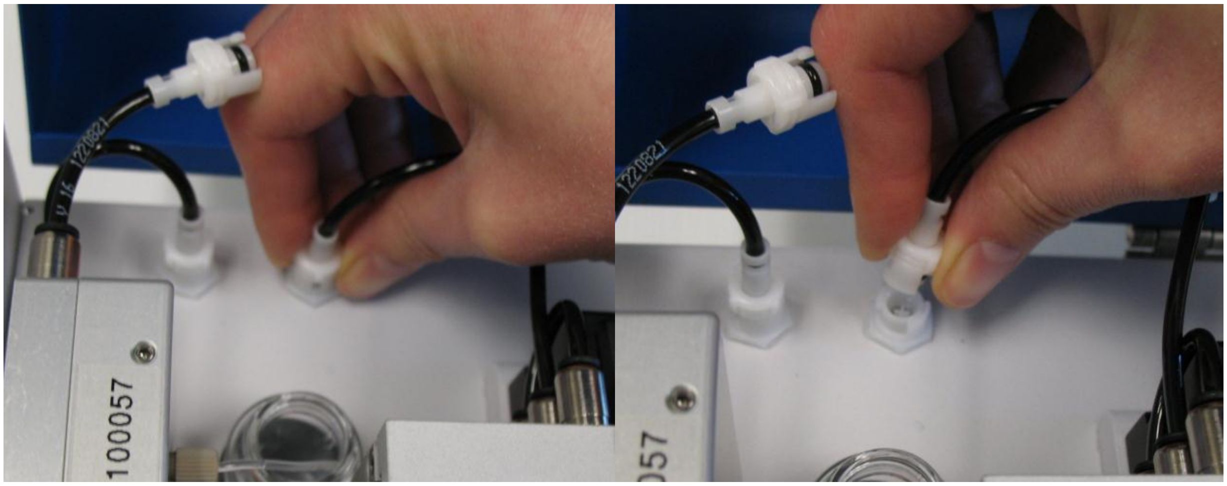
1. Make sure the valve to the VROC Cell assembly is closed, and the syringe jacket assembly is open.
2. Unplug the VROC Cell assembly:



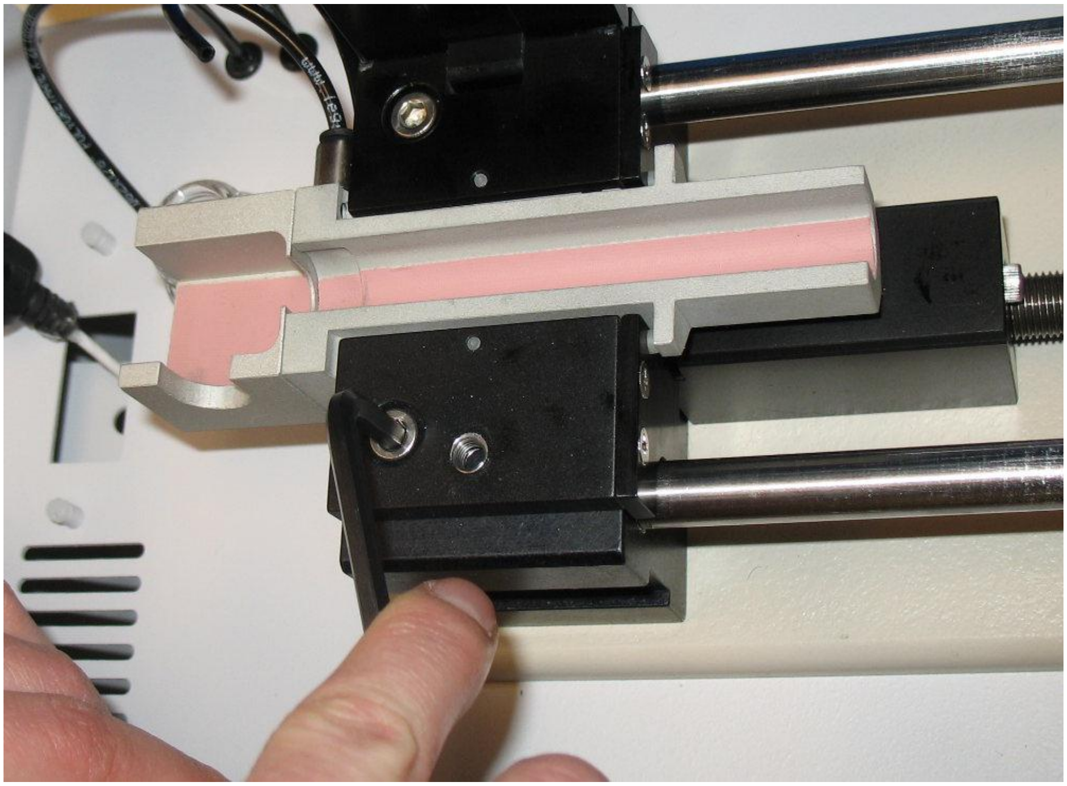
3. Disconnect the VROC Cell from the syringe jacket by firmly grasping the connection point, and twisting counter-clockwise:



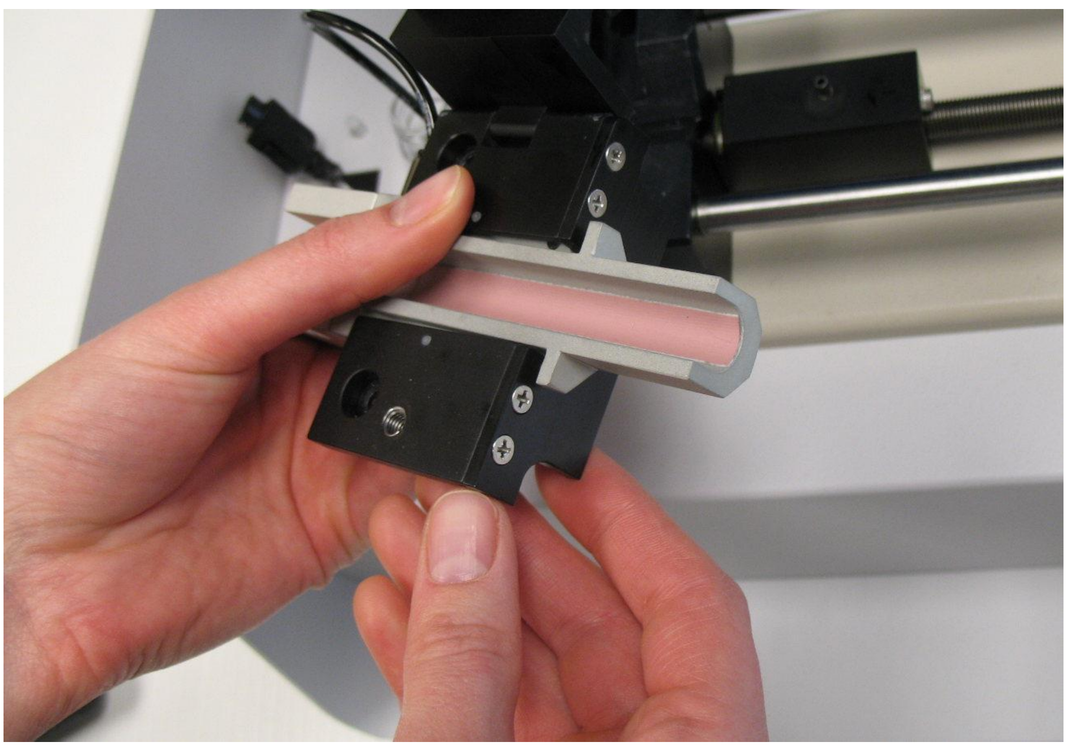
4. Disconnect the syringe jacket from the unit by grasping the white connector, and twisting counter-clockwise:



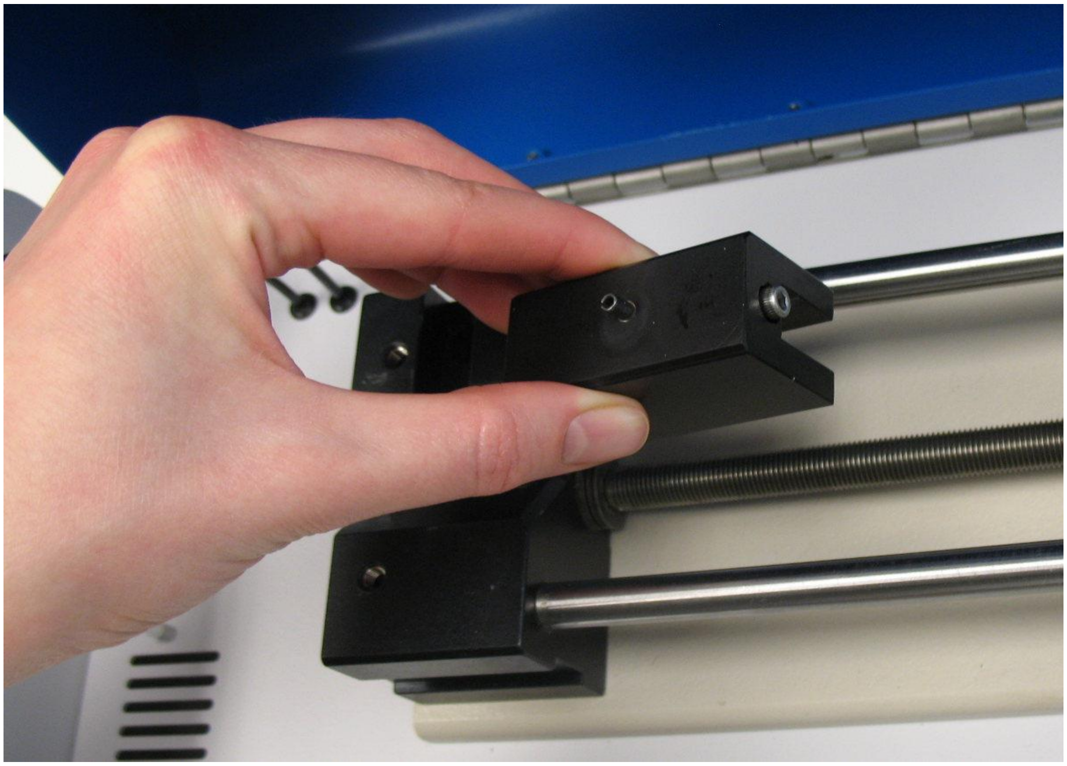
5. Using the 5/32" Allen wrench, remove the hex head screws in the bottom of the syringe jacket:



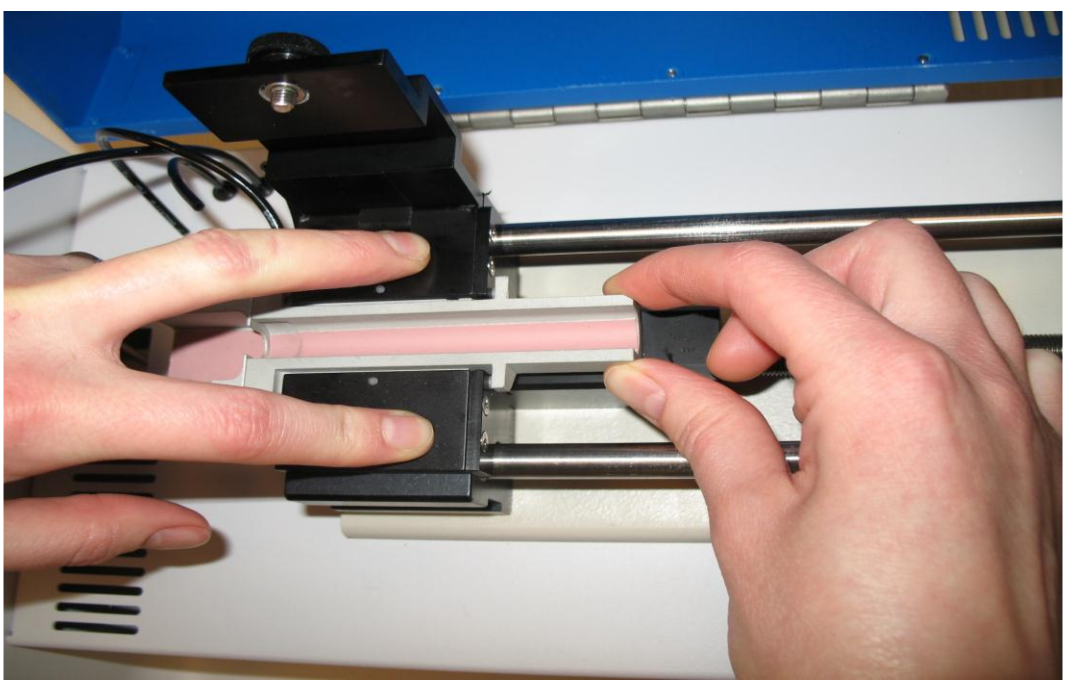
6. Remove the syringe jacket assembly from the *m*-VROC unit:



7. Remove the stopper block by lifting it out:

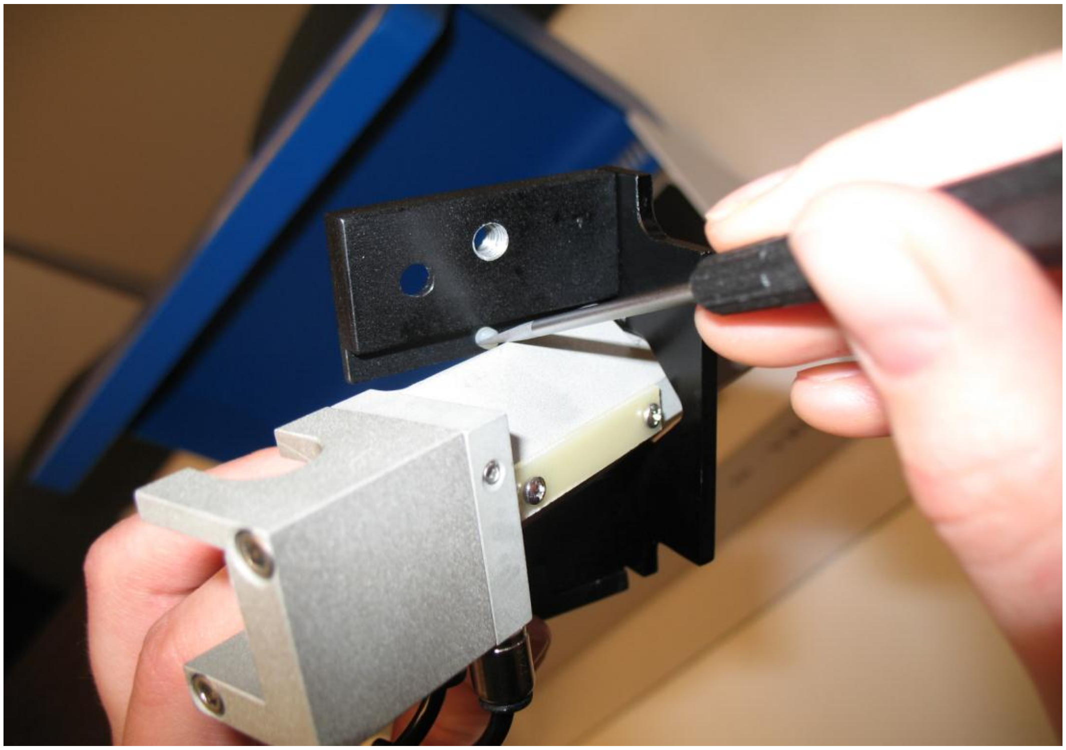


8. Select a new syringe jacket and its corresponding stopper block.
9. Put the new stopper block in place. Make sure the front of the stopper block is touching the back of the syringe jacket assembly cradle.
10. Place the new syringe jacket assembly in the cradle. Make sure the bottom of the jacket is pushed as far forward (toward the VROC Cell assembly cradle) as possible.
11. Simultaneously push down on the black "fins" and try to jiggle the bottom of the syringe jacket in the cradle:

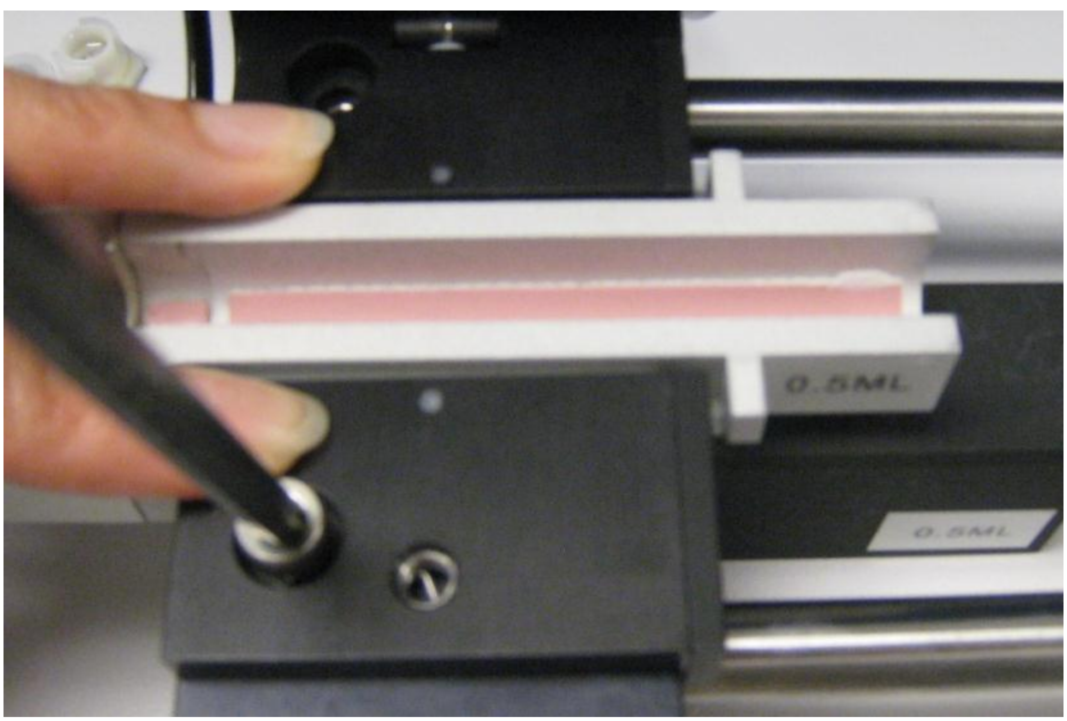


12. If the bottom of the syringe jacket stays in place, skip to step 14. If the bottom of the jacket moves, pull the syringe jacket assembly back out, and use a small flat-head

screwdriver to turn the small white adjustment screws one quarter turn, counter-clockwise. The heads of the screws are located on the underside of the black "fins":

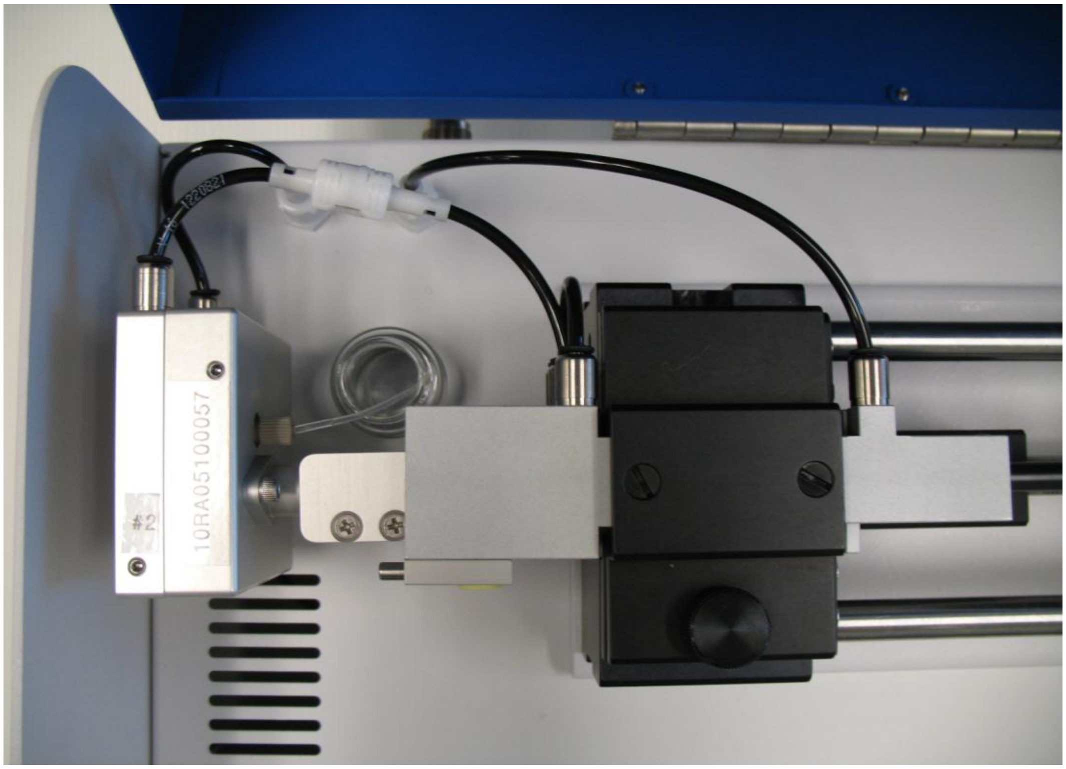


13. Repeat steps 11 and 12 as many times as necessary, until the bottom of the syringe jacket stays in place when doing step 12.
14. Place the hex head screws in the bottom of the syringe jacket assembly, and – while holding the assembly down – tighten them until snug:



15. Close the black "lid" over the top of the syringe jacket, making sure the thumb screw can be tightened (if it cannot, go back to step 10).

16. Re-connect the water tube from the top of the syringe jacket to the *m*-VROC unit (do the reverse of step 4).
17. Reconnect the signal cable and water tubes to the VROC Cell assembly. When the tubes to the VROC Cell are connected correctly, they will look like this:



D. Storing the Sensor

If the sensor will not be used for a month or more, and one or more of the sample fluids was a dissolved solution, it should be thoroughly cleaned before storage (this is not necessary if the sample fluids were pure solvents). This is because as the unit sits unused for a long period, the solvent will slowly evaporate from the exit tube of the sensor, and any solids that were dissolved will be left behind. These solids may become dried onto the sensor element, permanently damaging it. The procedure for preparing the sensor for storage is:

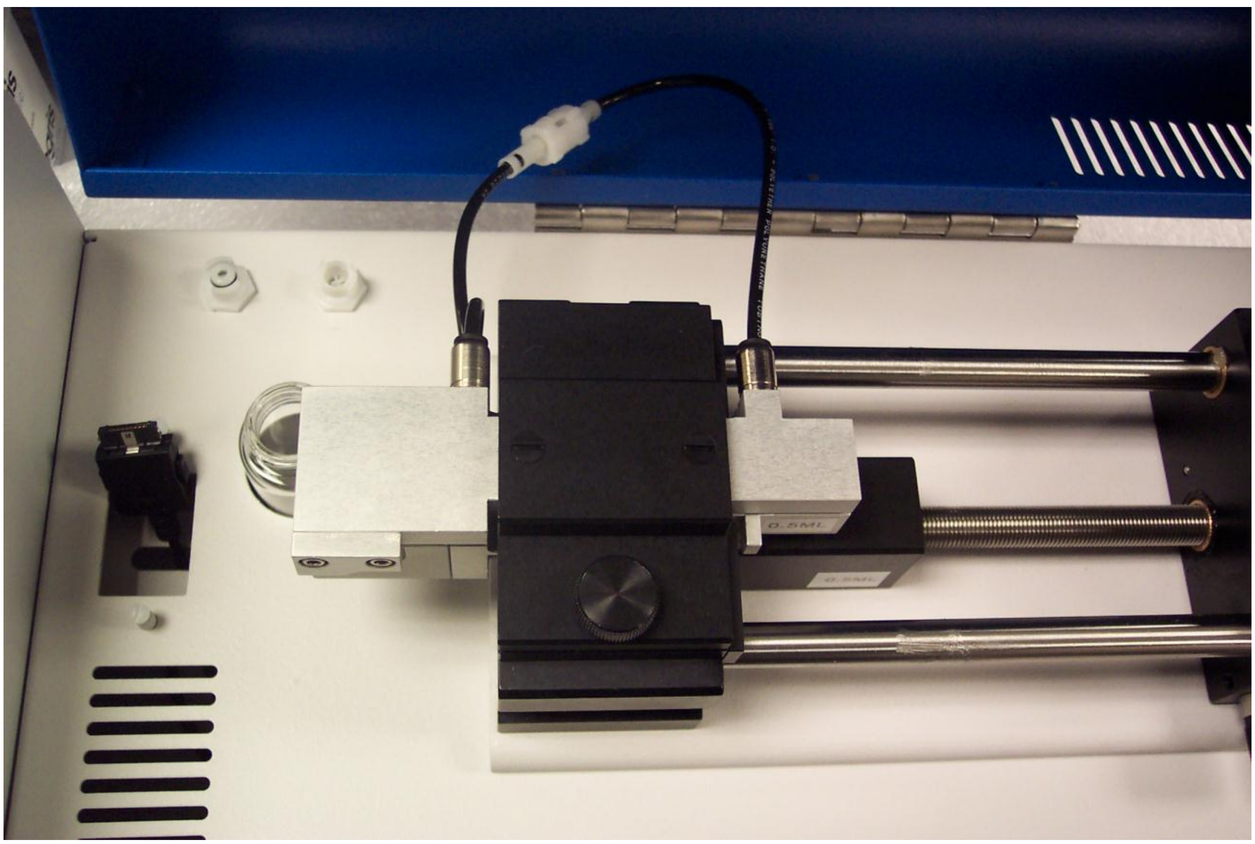
1. Clean the sensor thoroughly as described in section 7a.
2. Leave the sensor full of a pure solvent (such as IPA, heptane, or MEK), or full of a water solution with 1% Aquet detergent.
3. Close the valve on the VROC Cell assembly and remove the syringe from the valve block.
4. Install a threaded plug into the valve block in place of the syringe.
5. Disconnect the VROC Cell assembly from the *m*-VROC unit.
6. Connect the two white quick connect fittings to each other.

E. Storing the *m*-VROC Unit

If the *m*-VROC unit will not be used for a month or more, it is important to store it in such a way that any water in the temperature regulation channels inside the syringe jacket will not evaporate (deposits may be left behind that may clog the channels). In order to prevent this:

1. Disconnect the syringe jacket temperature regulation lines at the quick connects from the *m*-VROC unit (and the VROC Cell assembly if it hasn't already been disconnected).
2. Connect the two quick connect fittings from the syringe jacket to each other.
3. Do not store the *m*-VROC unit with a VROC Cell assembly in place, as the valve to the sensor cannot be closed.

The *m*-VROC unit will look like this:



8. Advanced Measurement Techniques

A. Temperature Control

When using the temperature control feature, it is important to keep the supply line from the water bath circulator as short as possible. The shorter the supply line, the less discrepancy there will be between the commanded temperature and the actual temperature of the liquid from the water bath circulator, and transitions between different commanded temperatures will be shorter. This is true even if using the closed-loop temperature control option. If the Thermocube water bath was purchased with the system, use the hoses that were included. Remember to use a dry air source to purge the system and close the top cover when taking measurements at temperatures below 18 °C; see section 4b for details on connecting the dry air source.

After a sample-filled syringe has been mounted, make sure to let the system come to full equilibrium before doing any measurements. Keep the lid of the *m*-VROC closed once the syringe is mounted, and while doing measurements; opening the lid can upset the equilibrium. The system will also take longer to come to equilibrium with larger volumes of sample, or if the commanded temperature is at either extreme of the operating range. Once the temperature of the sensor indicates that the system is in a steady state, it is a good idea to wait an additional 5 minutes before taking a measurement. The stability window feature in the Temperature Control/Measurement Advisor tab is a convenient way to do this. To verify that the sample temperature has reached a steady state, do a "dummy" run with a single shear/flow rate. If the temperature of the sensor moves away from the commanded temperature by more than 0.2 °C, stop the run and give the sample more time to reach the desired temperature.

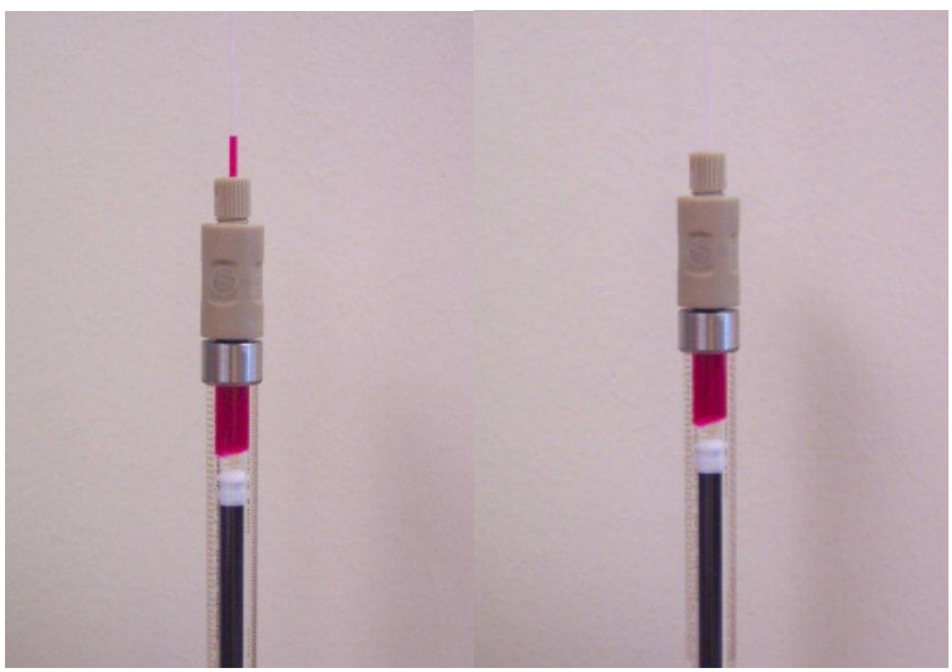
The default temperature of the Thermocube water bath is set to 25 °C by Rheosense. The default temperature may only be changed by using the front panel controls of the Thermocube. The *m*-VROC control program can send new temperatures to the Thermocube while in operation, but this does not change the default setting. Every time the Thermocube is turned on, it will begin driving the temperature to the default set point.

B. Alternate Sample Loading Method for Low Viscosity Samples (Front-End Loading)

With some low viscosity samples, it may be easier to load samples without bubbles using the following procedure rather than the procedure described in section 5a. This procedure is recommended for syringes of 1 ml or larger, and for fluids with viscosities of less than 400 mPa-s (cP).

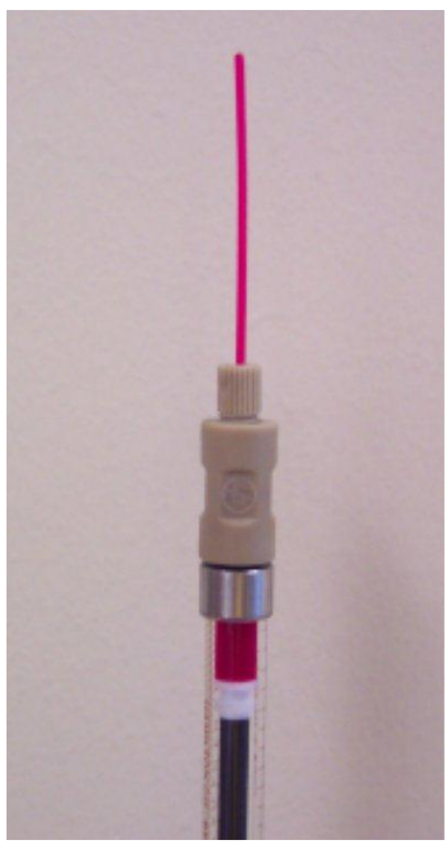
1. Leave the plunger a small distance from the tip of the syringe so that there is a small air pocket before withdrawing the sample. Slowly draw a small amount of sample into the syringe using the supplied plastic intake tube and union. Make sure no small air bubbles are generated:

2. Hold the syringe with the tip facing up and draw the sample completely into the syringe and out of the intake tube. Leave the air pocket at the bottom intact:



3. Tap the side of the syringe so the air pocket in the bottom rises to the top:

4. Push the air pocket out of the syringe and completely fill the intake tube with the sample:



5. Finish drawing as much sample as desired into the syringe, then load the syringe onto the pump as described in section 5b:

C. Loading High Viscosity Samples

It is quite difficult to load high viscosity samples (fluids with viscosities of 400 mPa-s or greater) into syringes without creating bubbles. This difficulty increases as the volume of the syringe becomes larger. The sample resists flowing into the inlet tube of the syringe when the plunger is drawn back, and a void (vacuum) may form between the sample and the end of the plunger. As atmospheric pressure pushes the sample into the syringe to equalize the pressure gradient, the sample fills in this void. This can take several seconds. In cases like this, there will almost always be a small bubble left attached to the end of the plunger. Bubbles in high-viscosity fluids will not be dislodged by the methods outlined in section 5a. Even a bubble on the order of only 1 µl in volume is generally large enough to adversely affect the measurement by significantly increasing the rise time. One way to ensure there are no bubbles loaded into the glass sample syringe is to back fill it.

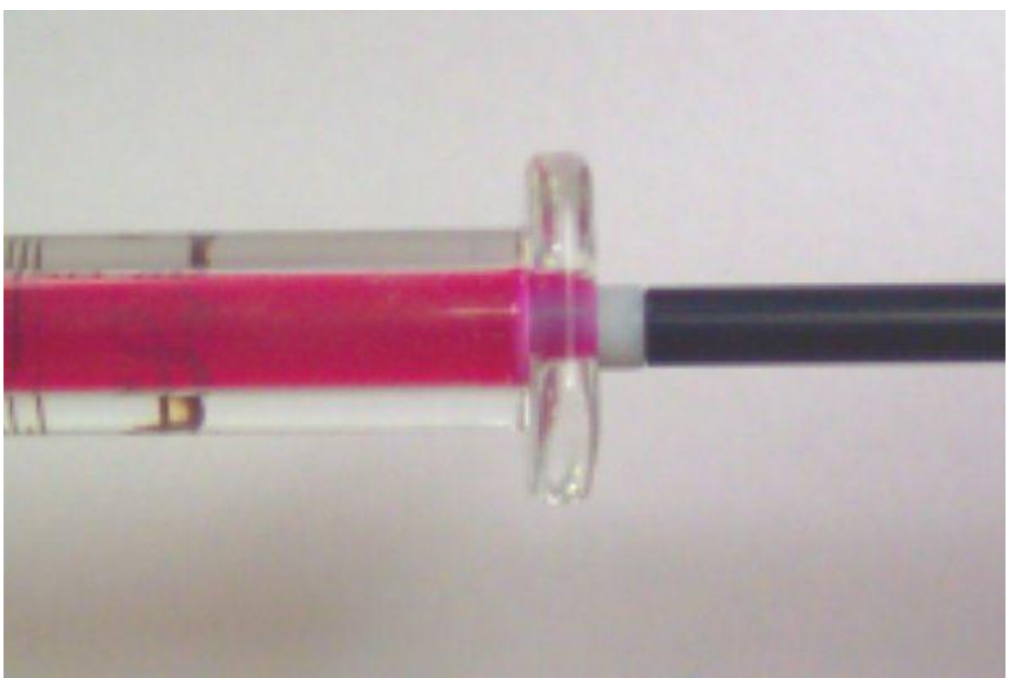
To do this, it is helpful to use disposable plastic syringes with a small diameter bore; a 200 µl syringe is a good choice.

1. Wet the plunger of the disposable syringe by drawing a small amount of sample into the syringe, then pushing it out. Do this several times until there are only small bubbles left attached to the end of the plunger that do not come off when the sample is pushed out. Fill the disposable syringe with sample and make sure that any bubbles present are attached to the base of the plunger only (not to any other place inside the syringe).
2. Remove the plunger from the glass syringe. Place the tip of the disposable syringe at the back of the glass syringe and slowly begin dispensing the sample into the glass syringe, making sure it is deposited without bubbles. Slightly tilt both so the tip of the glass syringe is down:

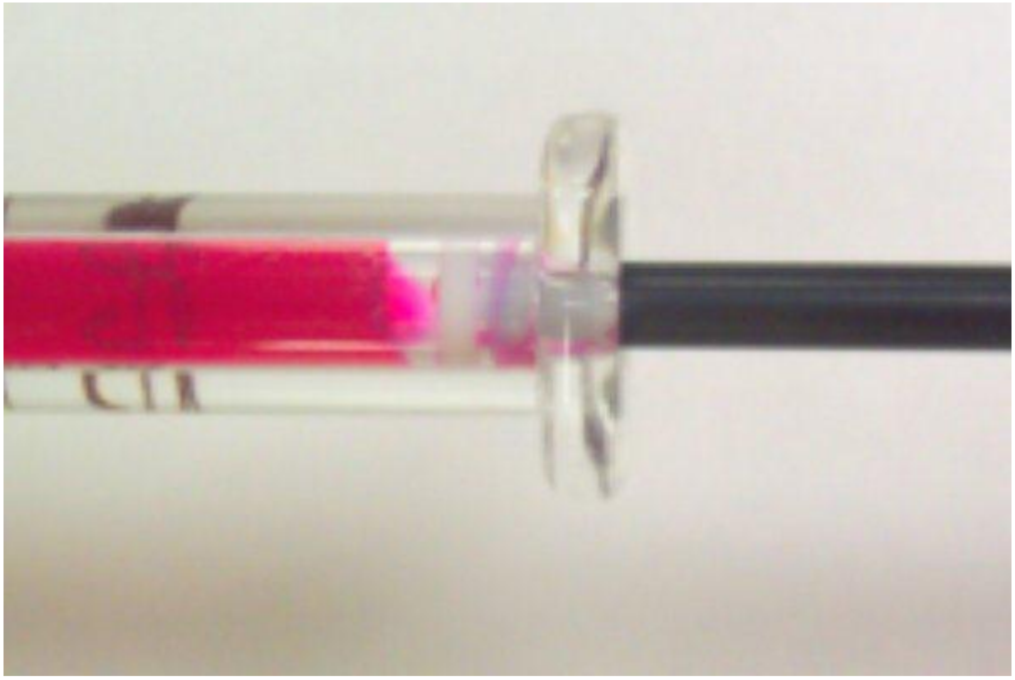
3. If the sample in the disposable syringe runs out before filling the glass syringe, load more into the disposable syringe and continue. Be careful not to introduce bubbles.

4. Wet the face of the glass syringe's plunger in the liquid at the top of the glass syringe, then insert the plunger carefully so that no bubbles are trapped:

Correct:



Incorrect:



5. Push the plunger in a little so that it is not too close to the end of the syringe. Load the syringe onto the pump as described in section 5b.

D. Testing Immiscible Samples

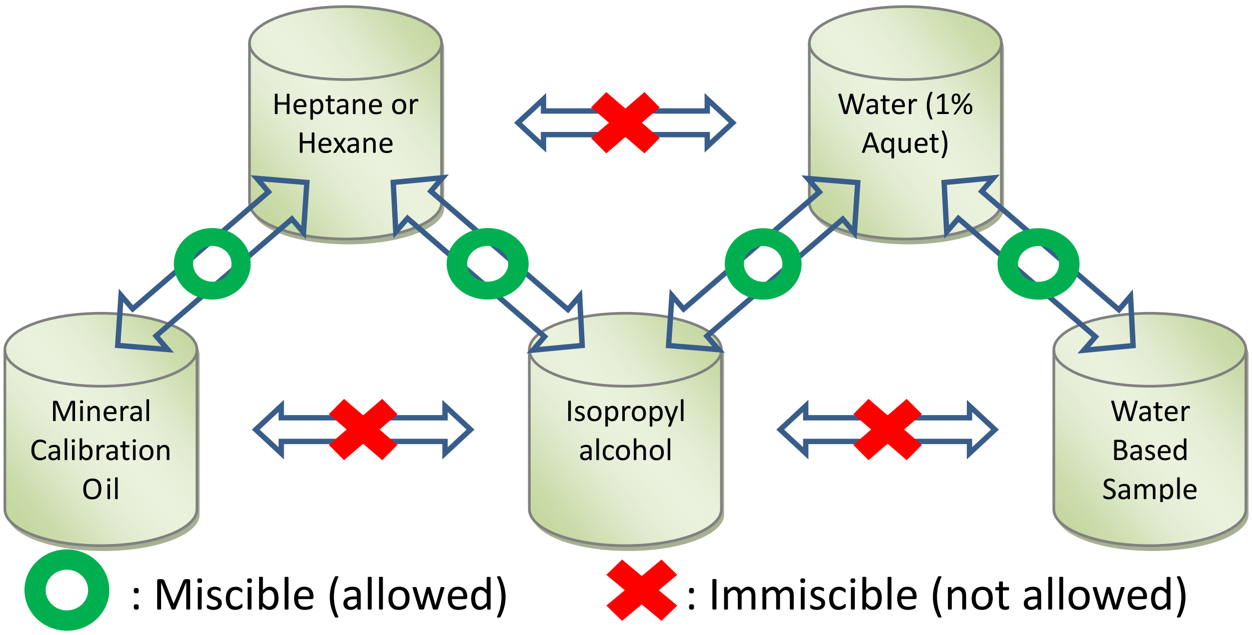
Consecutive measurement of two immiscible liquids with the VROC Cell can result in irreversible damage to the Cell, which may void the warranty. To prevent damage, a proper cleaning sequence is required when testing a series of liquids that are immiscible. The miscibility of liquids is not always obvious. For example, Cannon's standard mineral oil N400 is miscible with Heptane or Hexane, and Heptane is miscible with isopropyl alcohol. However, the same mineral oil is not miscible with isopropyl alcohol.

Check the miscibility of two liquids as follows:

1. Combine equal amounts of the two liquids in a small clear bottle or sample vial.
2. Vigorously shake or swirl the bottle until the two liquids are well mixed.
3. Let the solution sit for a minute or two and examine the mixture.
4. Miscible liquids should not have any visible phase separation, sedimentation or aggregates.

When testing such immiscible liquids, the sequence of exposing them to the VROC Cell is very important. For example, after checking the calibration of the VROC Cell with a standard mineral oil, a water-based sample is to be measured. The correct sequence would be: mineral oil, heptane, isopropyl alcohol, water (with 1% Aquet), water-based sample.

The correct sequence for this example is illustrated below by the arrows marked with a green O. To return to the mineral oil, the sequence would be reversed.



Appendix I

Troubleshooting Guide

Action being performed: initializing the unit		
problem encountered	**potential cause of problem**	**steps to resolve problem**
program freezes while initializing instrument, or error message pop-up reads: "Could not communicate with instrument. Make sure USB cord is plugged in then try again."	no power to unit	Check that the power cable is plugged in and the power switch is on.
		Check the fuse in the power entry module on the back panel of the unit.
	USB cord not connected properly	Check that the USB cord is plugged into the *m*-VROC unit and the computer.
	error in USB adapter	Unplug the USB cord from the computer, turn the unit off for 30 seconds, and close the *m*-VROC control program. Plug the USB cord back in, power on the instrument, and re-start the program.
		Re-boot the computer and try again.
error message pop-up reads: "Could not communicate with sensor."	USB cord not connected properly	Check that the USB cord is correctly plugged in to the unit and the computer.
	error in USB adapter	Unplug the USB cord from the computer, turn the unit off for 30 seconds, and close the *m*-VROC control program. Plug the USB cord back in, power on the instrument, and re-start the program.
		Re-boot the computer and try again.

Action being performed: taking a viscosity measurement		
problem encountered	**potential cause of problem**	**steps to resolve problem**
viscosity reading is not stable or is inaccurate	bubbles in the system	Tap the syringe to get the bubbles to the top, and expel a small amount of sample before loading syringe into the unit. Make sure to close the sample inlet valve before removing the syringe from the instrument.
		Do a "dummy" run to prime the system with a small amount of sample (35 µl or more) to push the bubbles out.
	VROC Cell is dirty or has not been properly flushed	Clean the VROC Cell with a compatible solvent as described in section 7a and Appendix II. It may be necessary to let the solvent sit in the VROC Cell for a few minutes. Confirm the VROC Cell is clean by measuring the viscosity of a known solvent (e.g., IPA).
	system was not properly primed when the baseline was measured	Do a "dummy" run to prime the system with a small amount of sample (35 µl or more).
		If the problem persists, contact RheoSense.
viscosity reading is not repeatable	faulty baseline due to residual pressure in VROC Cell channel from previous measurement	Increase the amount of time between measurements.
		Increase "Fall Time". See section 6d.
	"Pause Time" parameter not long enough to reach steady state reading	Increase "Pause Time" in the measurement input table.

<table>
<tr><td colspan="3">Action being performed: taking a measurement or monitoring the temperature</td></tr>
<tr><th>problem encountered</th><th>potential cause of problem</th><th>steps to resolve problem</th></tr>
<tr><td>temperature of the sensor goes above or below 25 °C before a test starts</td><td>default temperature of the Thermocube is not set correctly</td><td>Manually change the temperature of the Thermocube to 25 °C using the buttons on its front panel.</td></tr>
<tr><td rowspan="2">error message pop-up reads: “Error #### occurred at Error reported by sensor firmware: MEMS sensor error, ADC error, EEPROM error, Sensor board interface error.” where “####” is any number</td><td>sensor not plugged in correctly when m-VROC control program started up</td><td>Check that the cord to the sensor is properly plugged in so that it clicks, then click on the “Continue” button in the pop-up window.</td></tr>
<tr><td>sensor was unplugged while m-VROC control program was running</td><td>Re-connect the sensor, then click on the “Continue” button in the pop-up window.</td></tr>
<tr><td rowspan="2">error message pop-up reads: “Error 0 occurred at VISA Write in Take n Samples MVROC.vi-Read sensor loop for v3.vi-mVROC_Control v3.”</td><td>USB cable was unplugged while m-VROC control program was running</td><td>Re-connect the USB cable, then click on the “Continue” button in the pop-up window.</td></tr>
<tr><td>instrument was powered off while m-VROC control program was running</td><td>Power the instrument back on, then click on the “Continue” button in the pop-up window.</td></tr>
<tr><td rowspan="2">program freezes with no pop-up error message</td><td rowspan="2">error in USB adapter</td><td>Unplug all instrument USB cords (including water bath if applicable) from the computer and wait for about 30 seconds. Follow the directions on the pop-up window that appears.</td></tr>
<tr><td>Start the task manager and close the m-VROC control program.</td></tr>
</table>

Appendix II

Guidelines for *m*-VROC Cleaning

Low viscosity samples (Below 100 cP)	Medium viscosity samples	High viscosity samples (Above 1000 cP)
5 cc flushing	10 – 20 cc flushing	20 – 50 cc flushing

• Low viscosity inks • Low concentration polymer solutions • Various solvents	• Lubricants • Shampoo • Medium concentration polymer solutions	• Paints • High concentration polymer solutions

Appendix III

Selecting VROC Cells and Syringes

The *m*-VROC is offered in four VROC Cell specifications (A, B, C, and D) to maximize the viscosity range for precise measurements. The VROC - B Cell is our general-use viscometer.

The types of VROC Cells available are:

A Cells (A02, A05, A10)	Low viscosity (0.2 cP - 200 cP)	• Small sample volume • Low shear rate
B Cells (B02, B05, B10)	Medium viscosity (0.2 cP - 2,000 cP)	• General-use viscometer • Sufficient amount of sample required for low viscosity samples
C Cells (C02, C05, C10, C20, C30)	High viscosity (0.2 cP - 10,000 cP)	• Low viscosity samples: high shear rate, sufficient sample required • High viscosity samples
D Cells (D02, D05, D10, D20)	Highest viscosities (500 cP – 100,000 cP)	• Highest viscosity samples • Highest shear rates

To determine which VROC Cell to use for a specific viscosity or shear rate, use the interactive measurement advisor in the *m*-VROC control program. The measurement advisor displays the range of the connected VROC Cell, as well as the ranges of the Cells listed above. The ranges of VROC Cells can be shown in shear rate for a target viscosity, or in viscosity for a target shear rate. In addition, the software will suggest syringe sizes and minimum sample volumes to use for a given VROC Cell and measurement range. See section 6a-iv for a description of the measurement advisor controls. Contact RheoSense for further advice on VROC Cell selection for a given material or application.

Appendix IV

VROC Cell Specifications*

The flow channel width is 3.02 mm for all the following VROC Cells:

	Flow Channel Gap, μm	Maximum Pressure, Pa
m-VROC A-02	20	1.00E+04
m-VROC A-05	50	1.00E+04
m-VROC A-10	100	1.00E+04
m-VROC B-02	20	4.00E+04
m-VROC B-05	50	4.00E+04
m-VROC B-10	100	4.00E+04
m-VROC B-20	200	4.00E+04
m-VROC C-02	20	2.00E+05
m-VROC C-05	50	2.00E+05
m-VROC C-10	100	2.00E+05
m-VROC C-20	200	2.00E+05
m-VROC C-30	300	2.00E+05
m-VROC D-02	20	8.00E+05
m-VROC D-05	50	8.00E+05
m-VROC D-10	100	8.00E+05
m-VROC D-20	200	8.00E+05

* RheoSense, Inc. reserves the right to change specifications without notice.

Appendix V

MSDS for Aquet Detergent

PRODUCT IDENTITY: AQUET® DOUBLE STRENGTH CATALOG NUMBER (S): F17094-0020
EFFECTIVE DATE: 11-4-2008 MSDS NUMBER 01273 Rev 1 Page 1 of 4

1. Chemical Product and Company Identification

Catalog Number(s): F17094-0020
Product Identity: AQUET® DOUBLE STRENGTH
Manufacturer's Name: Bel-Art Products
Emergency Telephone Number (24 hr): 1-800-457-4280
Manufacturer's Address: 6 Industrial Road, Pequannock, New Jersey 07440-1992
Manufacturer's Toll Free Number/Fax Number: 973-694-0500 (1-800-4BELART)/973-694-7199
Internet: http://www.bel-art.com
2-25-2004, Revised 11-4-2008

2. Composition / Information on Ingredients

Percent Exposure Limits
Component CAS Registry # Concentration ACGIH TLV OSHA PEL
Ethoxylated Nonylphenol, branched 68412-54-4 39 – 41 N/A N/A
Dioxane 123-91-1 < 0.0006 20 ppm 100 ppm
Ethylene Oxide 75-21-8 0.0001 1 ppm 1 ppm
Water, Deionized 7732-18-5 Balance N/A N/A

3. Hazards Identification

EMERGENCY OVERVIEW
Does not present any significant health hazards. May cause irritation to eyes and skin. Wash areas of contact with water. For eyes, get medical attention.

POTENTIAL HEALTH EFFECTS:
TARGET ORGANS: eyes, skin
EYE CONTACT: May cause irritation, redness, and pain
INHALATION: May cause mild irritation of the respiratory system
SKIN CONTACT: May cause irritation, dermatitis, redness, and pain
INGESTION: May cause nausea, diarrhea, and abdominal cramps
CHRONIC EFFECTS / CARCINOGENICITY:
IARC – No
NTP – No
OSHA – No
TERATOLOGY (BIRTH DEFECT) INFORMATION:
No information cited in 'Registry of Toxic Effects of Chemical Substances' or other information sources.
PRODUCT IDENTITY: AQUET® DOUBLE STRENGTH CATALOG NUMBER(S): F17094-0020
EFFECTIVE DATE: 11-4-2008 MSDS NUMBER 01273 Rev 1 Page 2 of 4
REPRODUCTION INFORMATION:
No information cited in 'Registry of Toxic Effects of Chemical Substances' or other information sources.

4. First Aid Measures

In all cases, seek qualified evaluation.
EYE CONTACT: Irrigate immediately with large quantity of water for at least 15 minutes. Call a physician if irritation develops.
INHALATION: Remove to fresh air. Give artificial respiration if necessary. If breathing is difficult, give oxygen.
SKIN CONTACT: Wash areas of contact with soap and water. Call a physician if irritation develops.
INGESTION: Dilute with water or milk. Do not induce vomiting. Call a physician if necessary.

5. Fire Fighting Measures

FLAMMABLE PROPERTIES: FLASH POINT: 535-555 ºF METHOD USED: PM/CC
FLAMMABLE LIMITS: LFL: N/A UFL: N/A
EXTINGUISHING MEDIA: Use any means suitable for extinguishing surrounding fire.
FIRE & EXPLOSION HAZARDS: None known.
FIRE FIGHTING INSTRUCTIONS: None known.
FIRE FIGHTING EQUIPMENT: Use protective clothing and breathing equipment appropriate for the surrounding fire.

6. Accidental Release Measures

Collect liquid and dilute with water. Release to drain if local regulations allow. For larger spills, absorb with suitable material (vermiculite, clay, etc.). Collect the solid residue and save for disposal.

7. Handling and Storage

As with all chemicals, wash hands thoroughly after handling. Avoid contact with eyes and skin. Protect from freezing and physical damage. Store at 15 – 49 ºC (59 – 120 ºF) in tightly closed containers.

8. Exposure Controls / Personal Protection

ENGINEERING CONTROLS: No specific controls are needed. Normal room ventilation is adequate.
RESPIRATORY PROTECTION: Normal room ventilation is adequate.
SKIN PROTECTION: Chemical resistant gloves.
EYE PROTECTION: Safety glasses or goggles.

9. Physical and Chemical Properties

APPEARANCE: Free flowing, slightly viscous liquid
pH: 5.00 – 9.00 (10% Solution)
ODOR: Aromatic odor
BOILING POINT (ºC): > 200
SOLUBILITY IN WATER: Complete
MELTING POINT (ºC): - 4
SPECIFIC GRAVITY: App 1.06
VAPOR PRESSURE: Not Volatile
PERCENTAGE VOLATILES: 0.50% Water-Max

10. Stability and Reactivity

CHEMICAL STABILITY: Stable under normal conditions of use and storage
INCOMPATIBILITY: Strong oxidizing or reducing agents
HAZARDOUS DECOMPOSITION PRODUCTS: When heated to decomposition, can emit acrid smoke and fumes
HAZARDOUS POLYMERIZATION: Will not occur

11. Toxicological Information

LD50, Oral, Rat: 3000 mg/kg, details not reported other than lethal dose value.

12. Ecological Information

ECOTOXICOLOGICAL INFORMATION: No information found.
CHEMICAL FATE INFORMATION: No information found.

13. Disposal Considerations

Collect liquid and dilute with water. Release to drain if local regulations allow. For larger spills, absorb with suitable material (vermiculite, clay, etc.). Collect the solid residue and save for disposal. Always dispose of in accordance with local, state and federal regulations.

14. Transport Information (Not meant to be all inclusive)

D.O.T. SHIPPING NAME: Not regulated
D.O.T. HAZARD CLASS: Not regulated
U.N. / N.A. NUMBER: None
PACKING GROUP: None
D.O.T. LABEL: None

15. Regulatory Information (Not meant to be all inclusive - selected regulation represented)

OSHA STATUS: This item meets the OSHA Hazard Communication Standard (29 CFR 1910.1200) definition of a hazardous material.
TSCA STATUS: All components of this solution are listed on the TSCA Inventory.
CERCLA REPORTABLE QUANTITY: Not reportable
SARA TITLE III:
SECTION 302 EXTREMELY HAZARDOUS SUBSTANCES: No
SECTION 311/312 HAZARDOUS CATEGORIES: Acute: Yes Chronic, Fire, Pressure, Reactivity: No
SECTION 313 TOXIC CHEMICALS: No
RCRA STATUS: No
CALIFORNIA PROPOSITION 65: This solution contains an ingredient known to the State of California to cause cancer (Dioxane and Ethylene Oxide) and female reproductive toxicity (Ethylene Oxide).

16. Other Information

NFPA® Ratings: Health: 2 Flammability: 1 Reactivity: 0 Special Notice Key: None
HMIS® Ratings: Health: 2 Flammability: 1 Reactivity: 0 Protective Equipment: B (Protective eyewear, gloves)

Rev 1, 11-4-2008: (Section 16) revised health rating from 3 to 2.
PRODUCT IDENTITY: AQUET® DOUBLE STRENGTH CATALOG NUMBER (S): F17094-0020
EFFECTIVE DATE: 11-4-2008 MSDS NUMBER 01273 Rev 1 Page 4 of 4

When handled properly by qualified personnel, the product described herein does not present a significant health or safety hazard. Alteration of its characteristics by concentration, evaporation, addition of other substances, or other means may present hazards not specifically addressed herein and which must be evaluated by the user. The information furnished herein is believed to be accurate and represents the best data currently available to us. No warranty, expressed or implied, is made and RICCA CHEMICAL COMPANY assumes no legal responsibility or liability whatsoever resulting from its use.