Start-up Procedure

- 1. Verify that the air pressure is 5 bar or slightly above.
- 2. Check water level in circulator (only add DI water). Check water every week for cleanliness.
- 3. Turn on circulator pump.
- 4. If instrument is off turn instrument switch on. After a short start time device should say "Status: OK"
- 5. Remove foam bearing block or Teflon bearing guard. For foam block removal:
- 6. Press the "Online" button located on the front of the instrument to go offline for manual control.
- 7. Press the "Ref" button to move the bearing to the top position.
- 8. Push foam down to move block to the side.
- 9. After removal press "Online" button to go online again.
- 10. If the instrument was left on go to step 8.
- 11. Initialize the instrument. For initialization:
- 12. Open Rheoplus v3.40 software using the desktop icon.
- 13. Open the control panel by clicking on the icon of the instrument or through "Device"

 "Control Panel".
- 14. Click the "Initialize" button. This should be performed once per power cycle.
- 15. Allow electronics to stabilize for one hour (the Teflon bearing guard may be attached at this point and removed when the instrument is ready for the next step. This is important because the rotors expand while warming up).
- 16. Install the measuring system, making sure to match the lines on the measuring system shaft and the coupling.
- 17. In the Control Panel set the temperature to the desired setting. This switches on the Peltier temperature control system.
- 18. In the Control Panel set the zero gap by pressing the "Set Zero Gap" button.
- 19. Determine the inertia of the measuring system. For this determination:
- 20. Go to the "Service" tab of the control panel.
- 21. Press the "Meas. System" button.
- 22. Press "Start" button in the pop-up window.
- 23. After determination click the "Save" button.
- 24. Perform a motor adjustment. For this adjustment:
- 25. In the "Service" tab press the "Motor Adjustment" button.
- 26. Press the "Start" button in the pop-up window.
- 27. After adjustment press the "OK" button.
- 28. Press the "OK" button in the Control Panel and "Yes" when asked to save changes.

Procedure for loading and running emulsions

- 1. The larger measurement system is better for samples that easily flow. The smaller measurement system is better suited for samples that are more solid.
- 2. Shake the emulsion in the bottle to ensure sample being loaded is in suspension.
- 3. Use a disposable pipet to extract the emulsion from the center of the mixture to avoid any foam that may be on the top.
- 4. Pipet the emulsion onto the bottom metal plate.
- 5. Click "Lower measurement system." This is a button at the top right that looks like a downward pointing arrow next to a rheometer. A window will appear showing the progress of the lowering and then a second window will pop up once the trimming position has been reached.
- 6. If the sample is not very viscous (flows like water instead of mayonnaise) take the flat end of the spatula and scoop out any emulsion that has squeezed out (if no emulsion squeezed out around the measurement system more sample should be added). If the sample has too low of a viscosity to be trimmed in this manner, DO NOT dab it with a Kimwipe. Dabbing with a Kimwipe will take sample from under the plate. Be careful not to tap the measurement system while trimming.
- 7. Once you have trimmed and clicked "Continue," you should place the white plastic insulator gently around the plates. This insulation prevents any air drafts from drying out the sample as well as maintaining an even temperature.
- 8. Make sure that you set the temperature in control panel when you're doing anything besides a temperature sweep. If you want to perform the analysis at room temperature set it to 23 °C.
- 9. The sample loading is the same for all tests. We are now moving into how to do an amplitude sweep. An amplitude sweep is ideal for analyses if you're looking for shelf stability/the ability for the emulsion to stay in suspension. Generally, the lower the tan delta, the more likely it is to stay in suspension longer (this is directly related to a longer LVE range).
- 10. If starting a new template: When you click "Edit Meas profile" there should be 2 measurement points; the time unit should be in minutes; the profile should be in fixed measurement point duration; and the measurement point should be 1 per minute an interval of 2 minutes. A pre-shear and hold time should also be added on the measurements tab if starting a new template.
- 11. Next click "Start test" and name the sample. You should include the percent/amount of everything in your sample. For remark you should add any details that seem relevant.
- 12. In this same pop-up window go to the tab "Waiting before Starting Test" and make sure the wait time is set to 5 minutes. This ensures the sample heats/cools to the same temperature as the instrument. Then click "Start the Test Now."

- 13. Clean the plate and measuring system after each test (even if it's the same sample). Use a Kimwipe for the most of it. Next wet a Kimwipe with ligroin oil and wipe off any residue. Then use isopropanol (or acetone) on plate/measuring system and clean with a Kimwipe. Make sure it has evaporated before loading next sample.
- 14. You can calculate flow point and LVE range next if interested. This does need to be calculated if further oscillatory tests (such as a frequency sweep) are planned. Though generally, the current LVE range on a frequency sweep is good enough for most samples.
- 15. For the temperature sweep, add a wait time in the measurement window of about 10 to 15 minutes. The instrument takes a long time to change temperature.

Troubleshooting

- 1. If the sample freezes (for 10% wax emulsions, it's below 5 °C), a sharp increase in viscosity (table will have errors) and frost forming on the measurement system will be observed. Set the temperature (in control panel) to 23 °C before attempting to lift the measurement system. The Rheometer makes weird noises at different temperatures sometimes, don't be alarmed.
- 2. If there are large air bubbles on the plate, remove them using a pipet.
- 3. If there are a lot of peaks, that means there are either vibrations from the room or the emulsion is extremely unstable. Something as simple as bumping into the bench could cause a peak so it's best to leave the room entirely during tests.
- 4. If you drop the measurement system OR bump hard against the instrument, inform the Instrumentation Specialist your research advisor immediately. This could potentially ruin the instrument.
- 5. If you are trying to calculate LVE range but windows keep popping up about values missing, go to the white arrow on the top left and click that.

 Then click the first G' point and the second to last G' point on graph. It should now be highlighted. Then right-click, go to "Analysis" and then "LVE range". Make sure you delete error points before calculating LVE range.
- 6. To do calculations, select the current measurement for the graph of interest. Located on the left there is a pencil in the corner above all the samples. Click this pencil and the highlighted sample will be open for you to edit (unclick or click current measurement).

Shutdown Procedures

Procedure One:

When the instrument is to be used the next day:

- 1. Open the control panel by clicking on the icon of the instrument or through "Device" "Control Panel".
- 2. Under "Temperature," press the "Switch off" button to switch off the Peltier temperature control system.
- 3. Turn off or unplug the water circulator.
- 4. Remove the measuring system.
- 5. Attach the Teflon bearing guard.
- 6. Remember to shut off the air.

Procedure Two:

When the instrument will not be used for a while:

- 1. Remove the measuring system.
- 2. Attach the Teflon bearing guard or foam bearing block.
- 3. Turn off the instrument using the power switch.
- 4. Turn off the water circulator.
- 5. Remember to turn off the air.