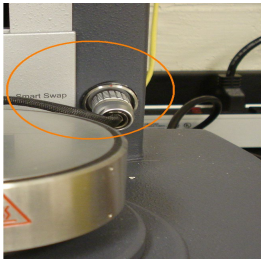


AR2000 Rheometer: Instructions

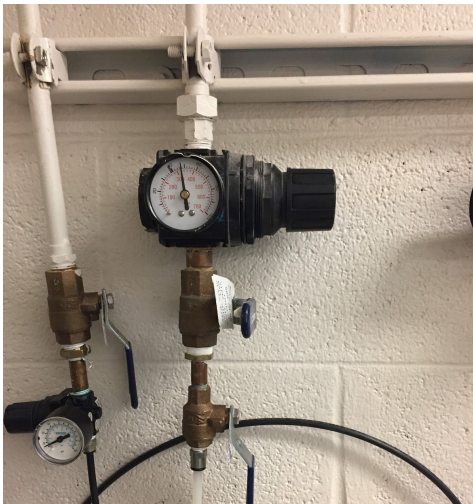
Instrument Setup

Note: The order in which the things are powered on is very important!

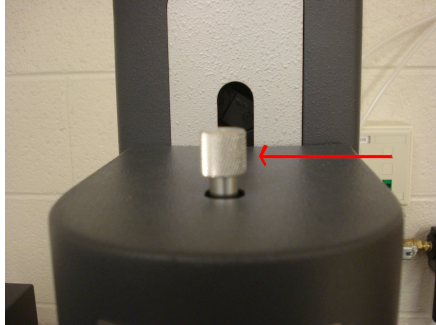
1. Check to make sure the Smart Swap cable is connected to the machine.



2. Make sure the compressed air line is open before turning on the instrument. This may take a few seconds as the pressure stabilizes to a constant value of 30 psi.



3. Remove the bearing lock cap by rotating the screw on the top in the counter clockwise direction and leave the screw as it is.



4. Turn on the water pump by placing the plug into the outlet.

5. Make sure that the inlet of the pump is immersed in water and the water level does not exceed the maximum level marked by the lower edge of the tape. When re-filling with water, add 1/4 of TA Conditioner bottle for heat exchange.



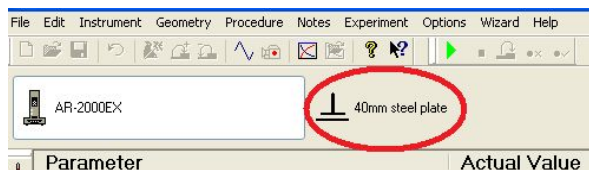
6. Turn the machine on. The power switch is located at the back of the controller on the right hand side.



7. At this stage, the spindle of the rheometer should be freely turning and the temperature reading on the monitor should be constant.

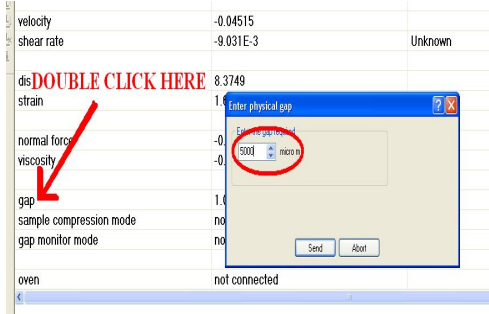
Software Setup


1. Click on the <AR Instrument control> icon on the desktop.
2. First, calibrate the instrument inertia. This only needs to be done ~once/semester and will be performed by the person in charge of the instrument. Go to the “options” tab on the top tool bar and select “instrument”. On the pop-up, go to the “inertia” tab, and select “calibrate”. Let the instrument calibrate, and the final value for the instrument inertia should be about 15 micro N.m.s².
3. Attach the appropriate geometry by gently pushing the spindle up the screw and tightening the top screw by rotating it clockwise. Click on the geometry tab and ensure that the proper geometry is attached. If the geometry name needs to be switched, go to the “geometry” tab and select “open”. From here, you can select the proper geometry that is being used.

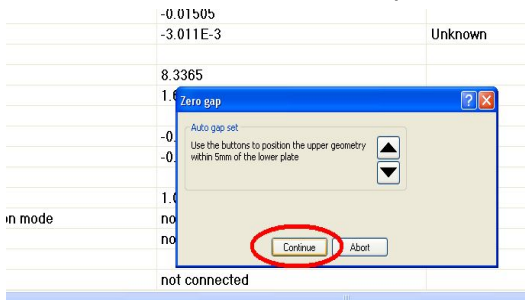


4. Now calibrate the bearing friction. Go to the “options” tab on the top toolbar and select “instrument”. On the pop-up, go to the “miscellaneous” tab and select “calibrate”. Let the instrument calibrate.
5. Click on the <instrument rotational mapping> button on the left hand side. This subtracts the background force that is used up by the air - bearing shaft to rotate about its own axis from the actual shear measurements. It is necessary to perform this when switching between geometries and performing low viscosity experiments.
6. Click “perform mapping.” The process takes about 5 minutes.

7. Double - click on the displayed value of “Gap”, enter 1500 micrometer and click on “send.”



8. Click on the < zero gap >  button. A dialogue box will appear on the screen. Click on continue. This makes sure that the Peltier plate and the geometry are perfectly parallel to each other.

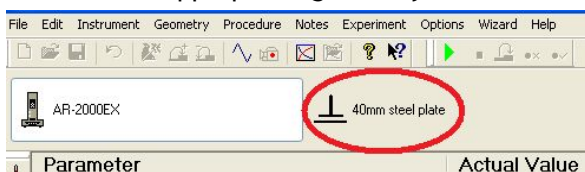


6. Once the zero gap procedure is completed, a message will appear on the screen asking if you would want to set the head to back off position. Click on “Yes.”

7. For Temp Sweep Experiments ONLY: Perform “Gap Temperature Compensation” at this point. See end of document for procedure.

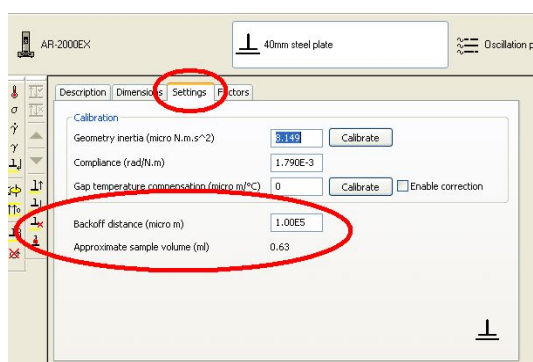
Loading of sample

1. Select the appropriate geometry tab below the task bar.



2. Click on dimension tab and enter the value of the gap between the upper geometry and the lower plate. The recommended gap value is 1000 microns (but this does vary from sample to sample). A typical value is 10x the largest particle size in your system.

3. Click on the “Settings” tab. Based on the gap value entered and the type of geometry, the software gives an estimate of the amount of the sample (in mL) that would be ideal for the experiment. It is a good idea to add slight excess.

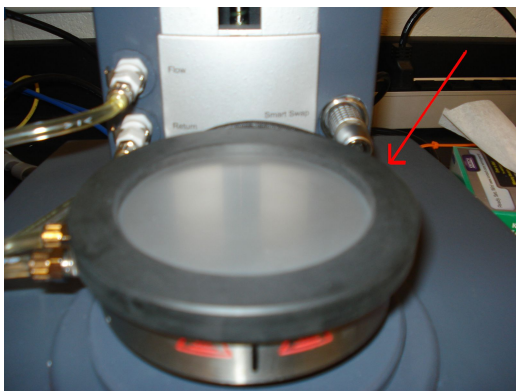


4. If the solvent is volatile, then it is recommended that you use the solvent trap for the experiments.

(a). Click on the bearing lock icon on the LHS column of the screen. This will prevent the head from turning. **CAUTION: DO NOT TRY TO TURN THE BEARING HEAD WITH THE BEARING LOCK ON OR YOU WILL DAMAGE THE INSTRUMENT.**

(b). Add the solvent to the back of the upper geometry (without turning the geometry - remember bearing lock is on).

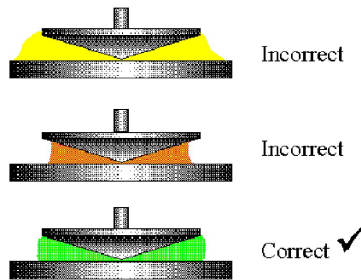
(c). Place the solvent trap ring in the middle of the Peltier plate.



6. Load your sample on the Peltier plate.



The most ideal loading of the sample on the plate is as shown in the figure. However one can overfill the sample and then trim off the sides.



Caution: In case of underfilling, discard the sample and load a fresh sample after thoroughly cleaning the Peltier plate and the geometry.

7. Send the gap to the truncation distance <Instrument> → <Gap> → <Go to geometry gap>.



8. Trim the excess sample from the sides.

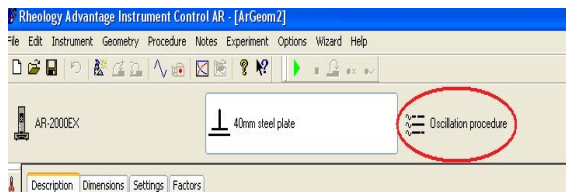
9. Add the solvent cover. (Make sure the solvent trap box is checked if you are using it.)



Taking Measurements

1. Make sure the instrument is set at the geometry gap (e.g., 1000 micrometers)

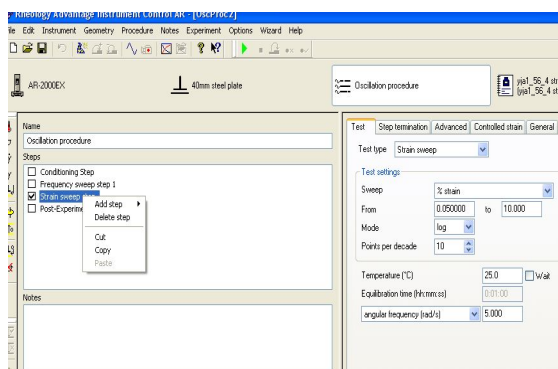
2. Set the oscillation procedure details by clicking on the “oscillation procedures” tab below the task bar.



3. Perform a strain sweep first to determine the linear viscoelastic region (LVR). This will allow you to determine an acceptable strain to use in the frequency sweep measurements. Load the sample and run the experiment.

4. Using a new gel sample, run a frequency sweep. Input the lower and upper limit of the frequency and the value of the stress you would like to use (that is in the LVR) (generally oscillation stress determined by the stress sweep experiments).

5. To add a step, right click on the previous steps and select add step.



6. Click on the “Play” button on the task bar. Input the sample information, file name and the directory in which it has to be stored.



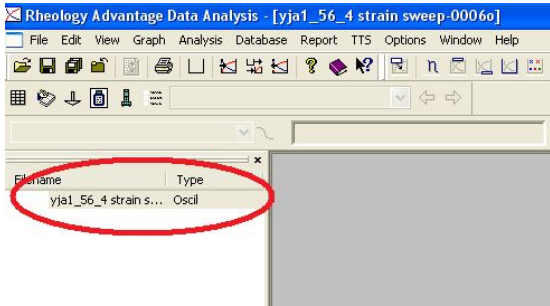
7. To take another set of readings, clean the Peltier plate and the geometry thoroughly with acetone (see section VI steps 1 and 2) and re-start from section II, step 4.

Data analysis

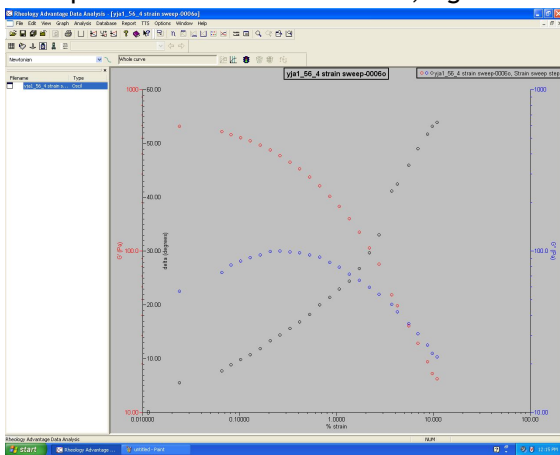
1. Click on the TA Data analysis icon on the Desktop.

2. Select the file from the location you stored it in section IV, step 5.

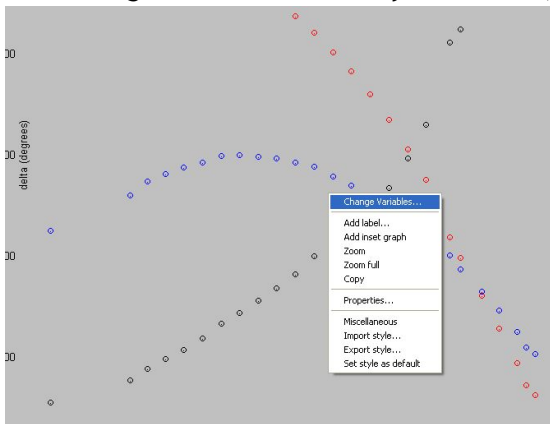
3. Double click on the file to get the graphical results.



4. To process data in the software, right click anywhere on the graph.

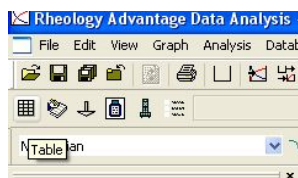


5. To change the variables on any of the axes, select the “change variables” option.



6. To change the appearance of the graph such as color, backgrounds, and the scale of the axes, click on “properties”.

7. Alternatively, to export data to Excel, click on the Table icon below the task bar. Right click anywhere to include more or less data variables. Copy the data into Excel.



Shut down

(The order of shut down is important!)

1. Send the geometry to the back off distance by <Instrument> → <Gap> → <Raise Head>. Remove the geometry by gently pushing it upwards and turning the screw clockwise.
2. Gently clean the Peltier plate and the geometry with acetone. Rheological measurements are very sensitive to impurities, so thorough cleaning between two consecutive readings and before shutting down is highly recommended.

Caution: While cleaning the Peltier plate, make sure minimum force is exerted on the plate as the instrument is sensitive to the amount of force applied on the Peltier plate.

3. Close all windows and running programs.
4. Switch off the machine by turning off the main switch at the back of the controller.
5. Switch off the pump and the motor by turning off the main power supply from the power strip.
6. Put on the bearing cap on the spindle similar to attaching a geometry.
7. Turn off the compressed air line. Since the valve connecting the air line to the rheometer is a bit sensitive, it is recommended that only the main line valve is closed.

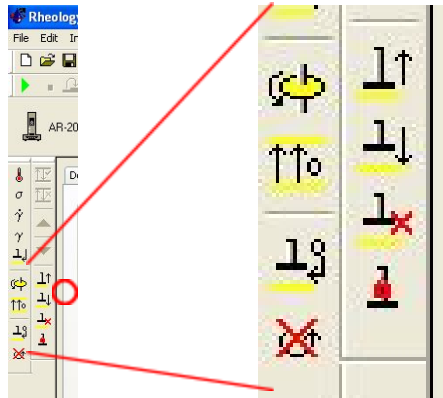
General precautions when using the heat exchanger

- The water pump should be running any time the Peltier plate is trying to control temperature (even close to room temperature).
- The water pump should be off any time the rheometer is off, or any time the Peltier plate is not connected or active.
- When operating at high temps, verify that the plate temperature is no more than 120°C greater than the circulator fluid temperature (e.g. if the fluid is at 20°C, the max plate temp is 140°C). At high temps it is also good to check the water level more regularly because it will evaporate more quickly. The water level should *never* be allowed to fall below the top of the pump.
- The pump should be oriented so that it is vertical, meaning that the water intake is from the top and the water intake be well submersed in water.
- Inlet and outlet tubings (blue tubes) should be free from kinks.

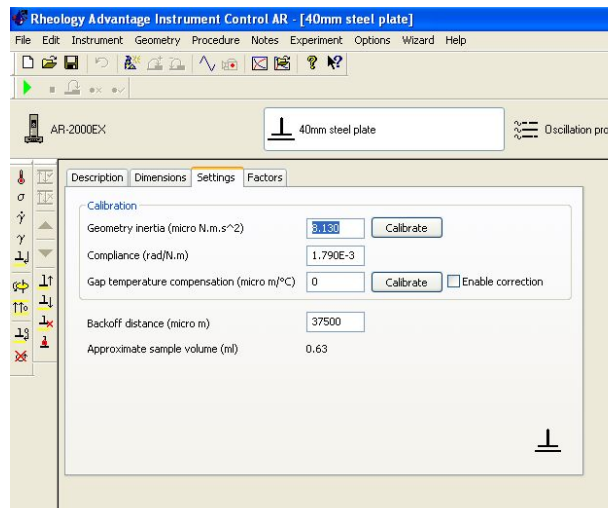
Procedure for Gap Temperature Compensation

To be performed the first time before loading the sample.

1. Lower the geometry to geometry gap by clicking on the ↓.



2. Click on the geometry tab -> settings and click on calibrate beside “gap temperature compensation”.

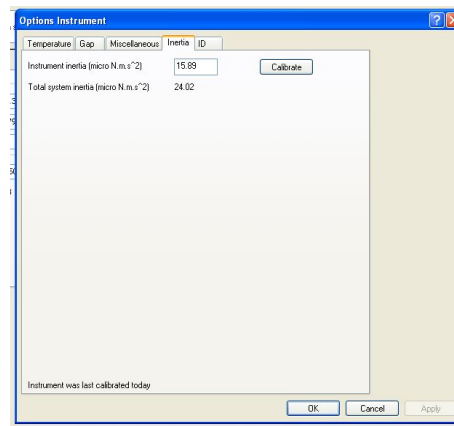


3. Enter the start and end temperature exactly identical to the temperatures in your procedure.
4. Click on “next”.
5. Finally, make sure “Enable correction” is checked.

Instrument Calibration

Instrument Inertia

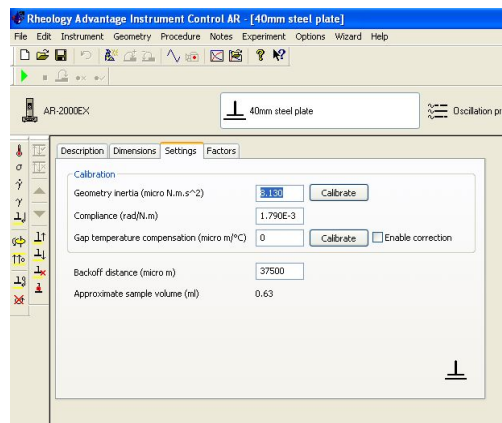
1. Go to “Experiment -> Instrument” and select “inertia tab”.
2. Click on “Calibrate”.



3. Make sure that geometry is NOT attached to the spindle and click on next.

Geometry Inertia

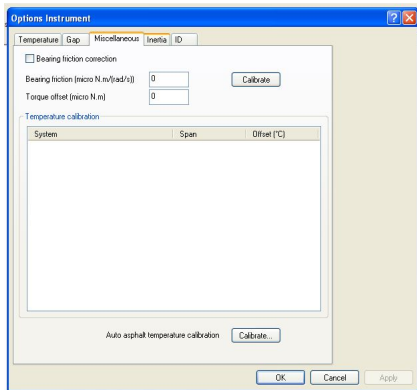
1. Attach geometry to the rheometer.
2. Click on the Geometry tab and click on calibrate.



3. Click on next.

Bearing Friction

1. Click on “Options -> Instrument”.
2. Click on Miscellaneous and click calibrate.



3. Click on Next.