Instructions for using the Anton Paar MCR 501 Rheometer

Instrument Summary:

The Anton Paar Physica Modular Compact Rheometer (MCR-501) applies a controlled stress (torque) to a sample and measures the strain (rotation). As such, the MCR is particularly well-suited to making measurements at very low deformation rates, which are typically required if the intrinsic response of microstructured materials is desired (higher deformation rates disrupt the microstructure). The MCR can access steady shear rates as low as 10^{-6} sec^{-1} and as high as 103 sec^{-1} and conduct dynamic oscillatory measurements as low as 10^{-4}rad/sec; cone-and-plate, parallel plate and Couette geometries are available, and temperature control can be achieved with a circulating water bath, a Peltier plate, or with an electric plate.

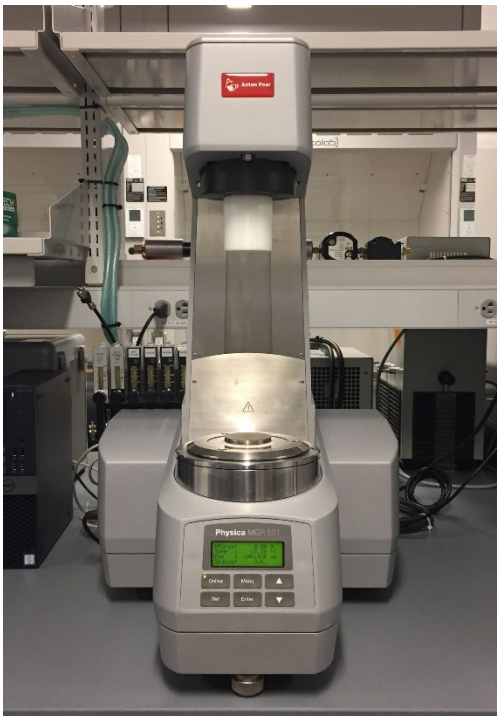


Figure 1. The Anton Paar Physica MCR 501 rheometer.

Safety and Precautions:

- **Warning –** Do not scrape the bottom plate. Only use Kimwipes to clean the instrument.
- **Warning –** Do not bump or contact the drive shaft located on the head of the instrument.
- **Warning –** Only transport the tools when they are sealed in their containers.
- **Warning –** Check the pH of all samples. Only use liquids with a pH in the range of 5-8.
- **Warning –** Zero the instrument in the configuration that will be used in the experiment.
- **Warning –** Only use the instrument if there is adequate N_2 pressure.
- **Warning –** Do not use PP (parallel plate) or CP (cone-plate) at shear rates larger than 1000 s^{-1}. In general, for high hear rates, use Couette or DG (double-gap).

I. Preliminary Instrument Inspection:

1. **Check N_2 Pressure –** Check the pressure on the valve located on the filtration unit. Pressure should be 60<P<100 psi. If the pressure is low, contact IAC staff and do not use the instrument.
2. **Check the N_2 Valve –** Make sure the valve located on the filtration unit is in the open position (pointed towards the plastic tubing).
3. **Check the Water Level in the Chillers –** Inspect the chillers ensure adequate water is in each tool. Add additional DI water if necessary.
4. **Ensure that the Accessories are Configured–** Ensure that the appropriate "accessory" (peltier plate, heated plate, CVT oven, cup/bob system etc.) is connected to the machine.

II. Setting up the Rheometer:

1. **Turn on the Chiller –** The Julabo water circulator will be used when performing experiments where the temperature is controlled. Flip the "Toggle Switches" located on the front and top-back of the Julabo circulator.
2. **Turn On the Rheometer –** Flip the "Toggle Switch" on the left side of the rheometer.
3. **Put the instrument in Online Mode –** Press the "Online" button located on the front of the instrument. This allows control of the instrument using the RheoCompass software.
4. **Open RheoCompass –** Click the "RheoCompass Icon", open the "Measuring Set" tab and select: Default MCR 501.
5. **Open the Control Panel –** Open the control panel by clicking the double-arrowed "Control Panel" tab located near the top-right corner of the RheoCompass window.
6. **Initialize the Instrument (if necessary) –** In the control panel, press the "Initialize" button. The instrument will raise the head of the tool to the reference point. Alternatively, the instrument can be initialized by pressing the "Ref" button while in offline mode.
7. **Select a Measuring Tool –** The tool inserts come in a variety of geometries: flat, cone, coquette, annular etc. Select the best geometry for your experiment.
8. **Remove the Teflon Safety Cap –** Carefully unscrew the white Teflon safety cap.
9. **Insert the Tool –** Carefully Insert the tool in the drive shaft.
    - Raise the quick-connect coupling on the drive shaft by lifting it directly up.
    - Match the black line on the tool-insert to the black line on the quick-connect coupling; gently insert the tool into the slot.
    - Lower the quick-connect coupling to lock the tool in place.

Note: Depending on the tool you select, it may be necessary to perform a motor and inertia alignment. This must be periodically performed for each tool every ~90 days.

10. **Configure the Instrument Using the Control Panel:**
    - **Reset the normal force –** Press the "Reset Force" button
    - **Set the Temperature –** Ensure that the Julabo circulator is on. Enter a temperature between ~5-100 °C, press "Set Value" (higher temperatures are available with additional configurations).

- **Set the Zero Gap –** Click "Set Zero Gap" to zero the gap between the base of the tool-insert and the plate. Watch the tool as it finds the zero point and raises. The normal force should briefly spike to 15-30 N and then drop back to 0 N as the tool begins to rise. Use the "Stop" button if the instrument fails to rise.

Note: Watch the motor when it is moving. Use the "Stop Button"  to prevent collisions.

11. **Move the Tool to the Loading Position –** Press the "Move to Loading Position" button in the control panel to raise the tool. *A standard loading position is ~60-80 mm.*

Figure 2. RheoCompass control Panel options.

III. Running an Experiment:

1. **Set the Measurement Parameters –** Use the "My Apps" page to design an experiment. Complete the process flow diagram by filling out the "Settings" tab.
   - **Pre-set Experiments** – There are a series of common experiments allowing you to adjust the stress, strain, frequency, and temperature to desired values.
   - **Custom Experiments** – Custom experiments can be designed from the "My Apps" tab by selecting "Flow Curve, Linear" and completing the flow chart.
2. **Place your Sample on the Plate –** Load your sample, follow the schematic below. Ensure that your sample is not corrosive. Do not scrape, scratch, or bump the plate in any way.
3. **Move the Tool to the Trim Position –** Press the **"**Move to Waiting Position" button to move the tool close to the plate. *A standard waiting position is ~5 mm.*
4. **Wait for Thermal Equilibrium –** Check the temperature readout. If necessary, use silicone oil or the environmental peltier hood (H-PTD) to limit evaporation of your sample.
5. **Move the Tool to the Measuring Position –** Press the "Move to Trim Position" button. *A standard measuring position is ~0.3-2.5 mm.*
6. **Trim Excess Sample –** For Cone-Plate and Plate-Plate tools, use a Kim Wipe to gently wipe away excess material.
7. **Start the Experiment –** Press the "Start" button at the top of the flow chart, then press "Play". Type in any experimental details and press "Continue" to start the experiment.

Note: Monitor the experiment when utilizing high speeds or temperatures.

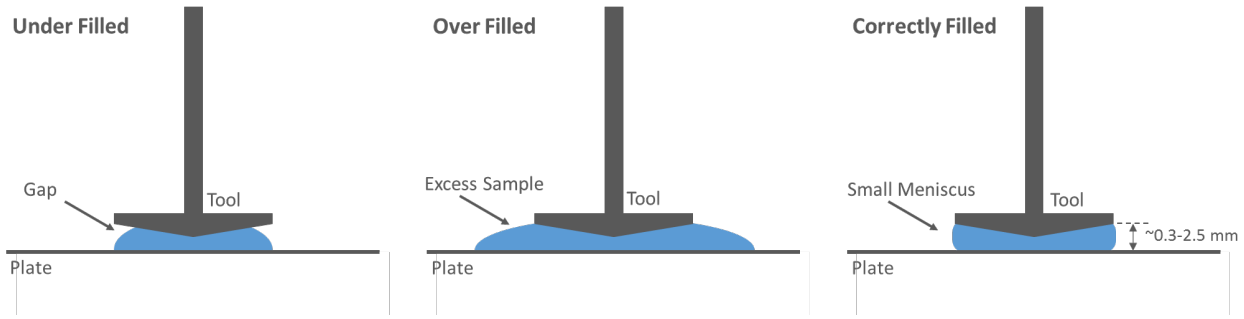


Figure 3. Schematic depicting properly and improperly loaded samples.

IV. Post-Experiment Cleanup:

1. **Equilibrate the Plate to Room Temperature –** Set the plate's temperature to 22 °C and allow the temperature to equilibrate.
2. **Tool Removal for *Adhesive Samples* –** For sticky or tacky samples, the tool should be detached from the motor before returning the tool to the loading position. This will prevent the motor from being stressed or pulled when raising.
    a. **Detach the Tool –** Hold the tool, lift up on the coupling ring.
    b. **Raise the Motor –** Continue holding the tool while pressing the "Move to Loading Position" button to raise the motor. Drop the coupling ring once motor stops.
    c. **Remove the Tool from the Plate –** Gently pull the tool from the surface of the gelled plate. Place the tool in the appropriate padded container.
3. **General Tool Removal –** For general Newtonian and non-newtonian samples.
    a. **Raise the Motor –** Press the "Move to Loading Position" button to raise the motor.
    b. **Remove the Tool –** Hold the tool, lift up on the coupling ring, remove the tool, slide the coupling ring downward. Place the tool in the padded container.
4. **Place the Teflon Cap in the Drive Shaft –** Screw the white Teflon cap into the head of the rheometer to protect the bearing and drive shaft.
5. **Clean the Tool Face –** *Only carry the tool while it is in the padded container.* Only use KimWipes, paper towels, acetone, soap and water, or other solvents to clean the apparatus. Do not use any caustics, abrasives, or excessive force to clean the tools. Do not allow the insertion end to get wet. Never wet the Tool Master interface.
6. **Clean the Lower Plate –** Use a KimWipe, and an appropriate solvent to *gently* clean off the lower plate.

Important: Do not scrape, scratch, or press on the plate with hard objects.

7. **Running Additional Samples –** If changing tools, always reset the normal force and zero the gap with the correct lower plate in place.

V. Shut-Down Procedure:

1. **Turn off the Temperature Control –** Press the "Switch Off" button in the control panel.
2. **Turn off the Water Circulator –** Flip the two "Toggle Switches" to the off position.

3. **Fill out the Log Sheet –** Sign the log sheet, list which tools were used, and any comments or observations noticed during the experiment.
4. **Log-Off the Instrument –** Close the software. Remember to log off the instrument.
5. **Instrument Inspection –** Ensure that the instrument is left in the proper configuration:
    - The white Teflon cap is covering the drive shaft
    - The N_2 flow is constant at $60 < P < 100$ psi
    - The N_2 valves are open
    - The temperature controller is off
    - Water circulator is off
    - The instrument is on
    - There are no scratches or spills on the plates and tools

Appendix

RheoCompass Software

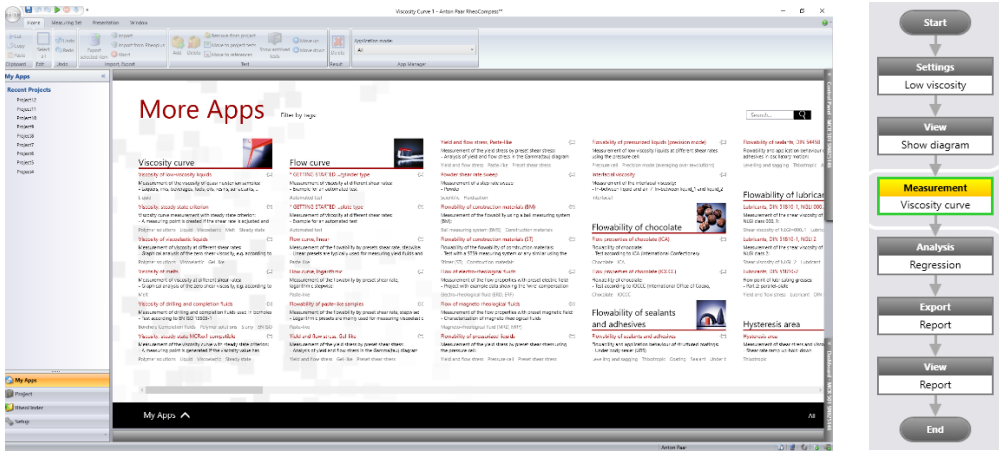
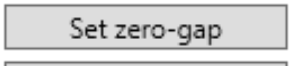


Figure 4. Pyris software window layout and process flow diagram.


Move Up

Stop Operation


Move Down



Set Zero Gap

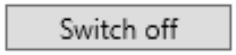
Reset Force


Move to Loading Position


Move to Waiting Position


Move to Trim Position

Set Value



Switch Off

- **What if Scenarios:**
  - **Unable to Connect with the Instrument –** Turn the Rheometer's toggle switch off, wait a minute, restart the computer, turn the toggle switch on.

  - **A Tool is Dropped or Damaged –** Dropping a tool could impair the accuracy of future measurements. Record the incident in the log book and contact IAC staff.

- **Available Tools and Configurations:**

  Geometries include parallel plates (25 mm, 50 mm, and 50 mm serrated), cone and plate (25 mm, 50 mm, and both at 2° angles), cup and bob (Couette cell), and double walled gap Couette (for annular flow). Accessories include a peltier plate (with both peltier and electrically heated hoods), electrically heated CVT 450 oven and a SER extensional device.

Safety requirements

The laboratory in room ACEE 027B is a wet chemistry lab, in which some researchers may handle hazardous substances. The lab safety requires that users wear **eye protection** (safety googles or glasses with side shields), **lab coats**, and **gloves** while using equipment in this room.

Emergency Information:

Medical Emergencies: Contact 911 and Public Safety (609) 258-1000
Room / facility emergencies: Contact Public Safety (609) 258-1000
Issues related to the instrument:

1. Contact IAC Staff.
2. If unsure, leave system as is.
3. Try to power down the system.

Audible/Siren Emergency Alerts:
Follow previous steps 2 & 3 and leave the building.

Emergency Contact Information:

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