Instructions for TA HR-2 Shear Rheometer

You must be authorized to use this equipment. To become an authorized user you must have your advisor email a request to Dr. Faith Morrison

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(rheo2016)

Note: There is no chemical storage in room 309A. Do not leave chemicals in the lab.
Glossary of Terms

The HR-2 consists of three parts: The test station (where the sample resides), the electronic unit, which houses the instrument control electronics, and the computer.

The front of the test station has a touch pad with the following buttons (from left to right):

1. up
2. down
3. zero gap
4. zero normal (trim gap)
5. lock bearing
6. start
7. stop
8. smart swap,
9. control panel
Instructions for TA HR-2 Shear Rheometer

(Authorized Users Only)

Installed 17 September 2013 by Michael Pepper from TA Instruments (Service Engineer Midwest, Phone 302-427-1028, office fax 302-427-4054, email mpepper@tainstruments.com)

Purchased by instrument grant written by Bruce Lee with contributing funds from the departments of BME and CM and from the CM4655 fee account (Morrison).

6 November 2013 Faith Morrison with input from Sarah Skelton


1 July 2015 Water flood from chemistry lab above: (functioning well)

10 July 2015 edits

3 December 2015: water circulator was allowed to run low—must be checked every time the instrument is run.

6 December 2015 FAM edits

Preliminaries and Log book

1. Safety glasses with side shields and appropriate laboratory attire are required in room 309 and 309A at all times, without exception. Violation of this rule will result in immediate expulsion from the lab.

2. This equipment is to be used by authorized users only. Please have your research supervisor contact Dr. Morrison to request for you to become an authorized user.

3. All users must sign into the instrument log book. Please sign your name, your contact information, and plans for tests into the log book. With shared equipment, it is essential that an accurate log book be maintained. Please note any difficulties or unexpected events that occur when you use the instrument. This information is essential for the continued good operation of the equipment. Please review the log book to be up-to-date on what the status of the instrument is.
Preparing to Run the HR-2

1. Verify the Air Bearing Shut-off Valve is open.
2. Verify the Air Bearing Regulator is delivering (32 psi) clean dry air.
3. Turn on the computer.
4. Unlatch the doors to the extended temperature cell and push the doors back
5. Remove the air bearing lock cap: this is a two-handed operation. Grasp the black lock cap with the right hand while holding the aluminum knob on the top of the motor assembly. Unscrew the lock cap and set it aside.
6. The power strip should remain on at all times. Turn on the electronic unit attached to the HR2 by toggling the power switch on the back of the unit. It takes several seconds for the unit to come online. When ready, the lights on the touchpad display will all cycle on leaving the START (green) and STOP (red) buttons illuminated.

Prepare the Software

1. On the computer double click on the TRIOS icon to load the software.
2. A dialog box appears indicating 5332-0199@NOT_SET and offering the Connect to Instrument. Click on Connect.
3. The software will connect to the instrument and open the File Manager, Experimental View, and the Control Panel.
4. On the test station LCD screen verify that the following number is displayed at the top:

   5332-0199:192.168.1.2

   The first 8 numbers are the serial number of this instrument; the second 8 numbers are the address of the COM port. If the LCD screen displays 000.000.0.0 the following has likely happened: the UP button has been pushed while the electronic unit is off. To fix this problem, proceed as follows: On the instrument touch pad, choose CONTROL PANEL, then select DOWN then hit START. The test station will reset and the COM port will be set correctly.
Install (or Remove) the Lower Plate (Peltier Plate)

Except when the Extended Temperature Cell is in use (ETC), the lower tool of this instrument is the Peltier Plate. The Peltier system controls the temperature of the sample being tested and provides the lower test surface. The Peltier Plate is installed via the TA Smart Swap system.

1. Install the Peltier onto the magnetic mount on the Test Station by aligning the alignment pin on the Peltier with the notch on the front of the mount.
2. Plug in the electronics of the Peltier by vertically aligning the red dot on the top of the (male) plug with the (female) receptacle on the test station. Push to connect.
3. Connect (align and push) the two Tygon tubing cooling lines into the quick-disconnect fittings on the test station (it does not matter which connection goes to which port).
4. Turn on the water circulator located on the floor behind the instrument bench. To turn on the circulator, flip up the toggle switch. The circulator should be on whenever using the Peltier Plate, which is suitable for temperatures between 5°C and 100°C.
5. To remove the Peltier, first turn off the circulator (flip down the toggle switch on the circulator on the floor behind the instrument bench). Then press SMART SWAP on the touch pad, which will then flash green; this releases the electronics. Now disconnect the water-cooling lines (the connections are quick disconnects; press on the metal release that is part of the quick disconnect assembly and pull out the line); then remove the electronics connection. Press SMART SWAP again and the light will stay green; this releases the magnet that holds the plate in place. The Peltier may now be removed from the Test Station. Press SMART SWAP again to return to normal operations mode.
6. In normal operations mode both the START and the STOP button lights will be on.

Circulator Maintenance

1. Check the water level in the circulator every time you operate and at least once per month.
2. Top off with distilled water if the water level is low.
3. If changing out the water then add 2 table spoons of conditioner.

Immersion Cell

The immersion cell is used to create a liquid or saturated vapor environment for testing. Install the immersion cell as follows:

1. Remove the lower Peltier and place the immersion cell onto the Peltier (ensure the O-ring is in the proper groove). The cut-out section goes over the water tubes. Finger tighten down the 3 nuts.
2. Reinstall the Peltier and zero gap.
**Installing the Upper Tool**

On the computer in the TRIOS software, in the Control Panel (on the right) go to Motor. The five icons in the Motor section are: STOP MOTOR, LOCK, ZERO DISPLACEMENT, START MANUAL OSCILLATION, and GO TO HOME POSITION.

The upper tool to be used depends on whether you are using the Peltier Plate or the ETC. For the Peltier Plate we own the following upper tools:

- 2°, 20mm diameter cone
- 20 mm diameter plate
- 8mm diameter plate

1. When installing the upper tool, the TRIOS software must be up and running.
2. On the software lower right side, select the motor tab. In this section, select the GO TO HOME POSITION button (looks like a gear).
3. Once the bearing lock starts blinking on the touch pad you are ready to install the upper tool.
4. The upper tool installs as follows: Align the vertical scoring on the tool to the scoring on the underside of the motor assembly. Screw in the tool until finger tight.
5. Wait 5 seconds. The Instrument will spin and read information about the tool (Smart Swap). Along the top of the software window the Geometry section will show the dimensions of the installed tool.
6. Verify that the cooling water is on and set the plate temperature as follows: Go to the Environmental tab, and set the desired temperature.
7. Calibrate for inertia, friction.
8. In the Control Panel, under the gap control section of the software select ZERO THE GAP. The tool will spin and the head will lower to find where the tool touches the plate. The software will ask if you would like to go to the loading gap (answer yes).
9. The next step is to map the tool to account for tool fine rotational motion (See page 9 of the *TA Introductory Guide to Using a DHR Rheometer*). This will determine the tool characteristics (the effort to turn, etc.). There are two types of mapping: rotational mapping (includes oscillatory testing) and oscillation mapping (more precise when used in oscillation mode). The choices are:

   - 1° precision, 7 minutes, every degree (use for viscosity <100 cp)
   - 6° standard (use for viscosity about 100 cp)
   - 10° fast (use for viscosity greater than 1000 cp)

Begin the rotational mapping by going to the Geometries tab, then double click on the geometry that is in-use, then go to the Calibrations Tab (lower left of central window), and choose Calibration under Rotational Mapping. Click on Calibrate.

10. Remember to remap when you change tools. If you re-install the same tool, after cleaning for example, you can skip mapping. If you change tool (geometry) you must re-map.
Removing the Upper Tool

1. After your test is completed, raise the geometry to the loading gap by selecting the icon under the gap control.
2. Unscrew the upper geometry, remove and clean.
3. To reuse the same geometry, select motor tab and select GO TO HOME, to reinstall the geometry.

Note: under Motor, GAP the icons are as follows: stop head, go to geo gap, go to trim gap, raise to loading gap, zero gap, zero axial force, head up heads down, options.
Planning the tests

Temperature Control

Temperature must be set at the time that the tool is zeroed. The tool will expand as temperature rises; the appropriate temperature/gap adjustment must be entered into the instrument. (how?)

Oscillation Mode: Small Amplitude Oscillatory Shear (SAOS)

Note: $2\pi$ radians per cycle; $\text{hertz} = \frac{\text{cycle}}{s} = s^{-1}$.

\[ \gamma = \frac{\theta}{\theta_0} \]  where $\theta_0$ is the cone angle and $\theta$ is the angular displacement of the cone (cone and plate)

\[ \gamma = \frac{R\theta}{H} \]  where $R$ is the cone radius and $H$ is the gap between the plates, and $\theta$ is the angular displacement of the upper plate (parallel plate)

SAOS is only valid if the data are taken in the linear viscoelastic (LVE) limit. To determine your sample’s LVE limit, first perform a set of strain sweeps. Monitor the quality of the sine wave with the figure on the right side of the screen. If the points do not look like a sine wave, they are not valid points for SAOS.

**Strain sweeps.** The goal of the strain sweeps is to determine how high a strain you may use without leaving the LVE region. It is suggested to perform strain sweeps at all frequencies that you will use in subsequent frequency sweeps (one frequency per decade). For example, if you plan frequency sweeps for $0.1 \text{ rad/s} \leq \omega \leq 100 \text{ rad/s}$, then it is recommended that you perform strain sweeps (each sweep with a fresh sample) at frequencies of 0.1, 1, 10, and 100 rad/s.

**Frequency sweeps.** The goal of frequency sweeps is to characterize the relaxation behavior of the material. The strains used must be in the LVE regime. The shear stress generated must be sufficiently high so that a valid signal is recorded. The difficulty lies in determining what strain is necessary in order to get a sufficiently high shear stress signal.

**Oscillation settings**

- Points per decade: n=3 or 4 are recommended; total number of points will be $n \times$ number of decades + 1
- Strain: controlled strain or xxxx or precision sampling? Lower torque limit?
- Motor mode: auto or xxxx
- Conditioning time: xxxx
- Sampling time: ssssss
- Step termination: yes or no
Flow Mode: Steady Shear flow

To perform start-up of steady shear flow with the TA Discovery, use Peak Hold and set up a run of the desired shear rate and duration. Data points may be taken as rapidly as 1/s.

Flow settings

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Starting the test

Recommended sample thicknesses for parallel plate operation are between 0.1 and 2\text{mm} gap. The cone tool has a fixed truncation gap of 52\text{\mu m}. The trim gap should be greater than this, perhaps 100\text{\mu m}. The trim gap and geometry gap are set in the Experimental window (if you do not set these gaps in your experiment, the most recent values will be used, which may not be appropriate for you).

1. Verify that the cooling water is on and set the plate temperature as follows: Go to the Environmental tab, and set the desired temperature.
2. Load the sample as follows:

   a. Go to the desired test temperature.
   b. Zero the gap (press ZERO GAP).
   c. Press RAISE TO LOADING GAP
   d. Place the appropriate amount of sample on the lower plate. **WARNING:** NEVER touch anything metallic to the Peltier plate. Use a wooden tongue depressor or other wooden tools. Avoid putting your fingers in the gap.
   e. Press GO TO TRIM GAP. Note that the TRIM GAP light on the touch panel will flash red when the normal force is too high; when normal forces have relaxed sufficiently, this light will turn green.
   f. Trim the sample
   g. Press GO TO GEOMETRY GAP (do not re-trim); sample is now loaded. See note above about normal force warning lights.
3. To start a test, set up a Procedure by clicking on the Experiments tab (lower left). A conditioning step may be added at the beginning of a test to allow the sample to come to thermal equilibrium or to put a desired amount of pre-shear on the sample.
   - Oscillation = Small amplitude Oscillatory Shear (SAOS, \(G', G''\))
   - Oscillation Ramp can be a frequency sweep or a strain sweep
   - Flow = steady shear flow (\(\eta, \Psi_1\))
   - Flow ramp can be a shear rate sweep or a shear stress sweep
   - To start, push Δ(start)
4. To manipulate the graph that displays on the instrument do the following:
   a. Select EDIT, SELECT, choose variables to plot
   b. Select CURVES, select Use Symbols

5. To export the data, in the Results section right click on the test (left panel) and choose to
   EXPORT and choose Excel. It is convenient to use the mouse to grab the data from the Excel
   screen and save all data together into one spreadsheet.
Turn off Rheometer

1. Remove all testing tools (cone, plate) and return them to their storage cases.
2. Remove the Peltier and store in its storage case. Turn off the circulator.
3. Close out the software.
4. Turn off the power to the electronic unit.
5. Install the bearing lock cap (this is a two-handed operation). Grab the bearing lock cap in your right hand and grab the aluminum knob on the top of the motor assembly. Gently screw on the cap.
6. Leave the air bearing air flowing at 32 psi.
7. Close extended temperature cell doors and latch shut.
8. Verify that the circulator is off.
9. Close the air ventilation duct (on the wall) to save energy.
10. Turn off the computer and clean up the area around the instrument.
11. Sign the instrument log book to indicate any problems or issues encountered during operation.
References

“TA Introductory Guide to Using a DHR Rheometer,” PDF supplied by TA Instruments-Waters LLC, 159 Lukens Drive, New Castle, DE 19720, October 2013. Available at: www.chem.mtu.edu/%7Efmorriso/cm4655/TAInstruments/2013TAIntroGuidetoDHR.pdf
