GPC Instructions

Amanda Higginbotham Tour Lab Created Sept. 2006

RUNNING SAMPLES

- 1) Prepare sample with ~20 mg of polymer in 10-mL of spec grade THF
- 2) Fill GPC vial ~3/4 of the way full with dissolved sample and seal with vial caps using special red crimper tool.
- 3) ***Also prepare 2 rinse vials that contain only fresh THF***
- 4) Switch GPC from Idle to Run Mode
 - In the PL-GPC 220 Control program, push the "Run" button under the mode box

5) Open the DataStream Monitor

- Click the DataStream Monitor icon on the desktop
- Under Channel menu, hit Add Channel
- Click "Data Stream 1 on COM2" in the left-hand column, and the select RI from the right-hand column
- Hit OK
 - You should now see the GPC response line moving as a function of time

6) Autozero/Purge the column

- Again, under the PL-GPC 220 Control program, hit the "Autozero" button located in the RI Detector box
- Wait ~1 min. (check under DataStream Monitor to see if line has stabilized)
- Hit the "Purge" button in the RI Detector box
- The system will now purge for 10 minutes.
- Hit the "Autozero" button again and wait until the signal response has stabilized to a flat line for at least 10 minutes. (This can take up to 30 min to 1 hr, depending on the system)
- Proceed to the next step while you are waiting for this to occur.

7) On the desktop, open the progam "GPC Online"

- Open an existing workbook; for simplicities sake, choose the one titled "Amanda"
- Click OK
- If an error message occurs, ignore it.
- Click the "Runlist" button on the left-hand side of the window
- Under the Runlist menu, choose "Add Sample"
- Enter sample parameters as follows:
 - a. Batch Name: enter the date in the following format: MM-DD-YYYY
 - **b.** Sample name: identify your sample however you wish
 - **c.** Filename: Leave blank
 - **d.** Sample Type: Unknown
 - e. Method: "View Only" \rightarrow Fill Down
 - **f.** the other three columns then fill themselves in automatically
- Repeat process from "Add Sample" for each sample you are running

- 8) In the GPC solvent chamber, remove waste return tubing and collect waste THF in a beaker
- 9) Set Autosampler Parameters
 - Back in the PL-GPC 220 Control window, click the teal "Auto" area under the Configuration box
 - Depending on the number of samples you are running, set the First Vial and Last Vial numbers, and mark them as Rinse Vials
 - i.e. if you are running 3 samples, the first vial will be 1 (a rinse) and the last vial will be 5 (also a rinse)
 - Double check this by looking at the graphic of the autosampler on the screen. The yellow circles represent actual samples to be tested while the blue circles represent the rinse vials. The gray circles are not in use, while the white circle is the zero vial and should NEVER be removed.
 - Hit "Send"

10) Insert actual samples in the autosampler as you have named them in the Run List

- i.e. Sample No. 1 will be the first sample run after the first rinse vial, etc.
- Be sure to place your vials in the correct order in the Autosampler!!
- Check to make sure your vials are placed between the 0 and 10 markers
- Close the Autosampler drawer all the way
- 11) When the DataStream baseline is flat and all software parameters are set, push the "Autosampler (OFFLINE)" button in the Autosampler box (under the PL-GPC 220 control window) to begin the run.
 - You should then see the button change to "Autosampler (ONLINE)"
 - Each sample will take 25 minutes to run
 - You will know when the runs are complete when this button returns to "Autosampler (OFFLINE)"

WHEN THE RUN IS COMPLETE

12) Switch GPC from Run to Idle Mode

• In the PL-GPC 220 Control program, push the "Idle" button under the mode box 13) Return the solvent back to recycle

This MUST be done before you leave for the day or else the column will run dry

DATA WORK-UP

14) Close the "GPC Online" program

- **15)** Open the "GPC Offline" program (from icon on desktop)
 - Open the same workbook the run(s) was saved under (i.e. Amanda)
 - Again, ignore the error message that may or may not pop up
 - Click "Runlist" button on the left-hand side
 - Double click the date the runs were made on (i.e. Batch Name)

- The individual samples should pop up
- Choose a Method (Double-click in the method box of the first sample, choose "Amanda", then hit "Fill Down")
- Highlight the first sample by clicking on the sample number button (a right arrow will appear)
- Once highlighted, click "Start Analysis" under the Processing menu
- To view the GPC trace, hit the "Analysis" button to the left of the window
- Delete peaks that are not contained within the high and low limit (this is set by the calibration curve)
 - **a.** Do this by hitting the "Peak Summary" tab and highlighting/deleting the extra peaks
 - **b.** Once you are happy with the peak(s) that have been selected, choose "Calculate Results" under the Processing menu
 - c. The Mp, Mn, Mw, Mz, and PD will then appear in the Peak Summary tab