GPC DMF – Manual
Polymer Facilities - MRL @ UCSB
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Log into FBS to access instrument. No reservations needed.

SAMPLE PREPARATION: Polymer **must be perfectly soluble** in DMF. Prepare solution 6-8 mg of the polymer in 2 mL of DMF with 0.01% LiBr (premixed solution is in the lab). Sample concentration affect both viscosity and injection volume. While small sample amounts produce narrower peaks, viscous samples may require larger, more dilute solution. After filtration through 0.45 µm filter, transfer solution into the 2 mL autosampler vial (fill between the second and third mark). Small volume inserts (150 µL) are also available.
NOTE: Sample intensity will be less and peaks will be broader in DMF. Also, PDIs may be larger than expected.

- Columns: 2 Tosoh TSKgel Super columns + guard (1 HM-M and 1 AWM-H)

STARTING EMPOWER SOFTWARE

1. Open Empower icon on desktop (login: user password: polymer).
2. Click on Run Samples
3. Click Chromatographic System MRL_DMF and Project Folder GPC DMF. Click on Ok.
4. Load sample set (use "Unknown Samples DMF mini 2414")

ADD SAMPLES to the running GPC (only red icon is active):

1. Edit- **Alter Running Sample**- OK. Red and green icons should be active.
2. Insert rows, change sample name, vial #. Don't forget to load carousel with vials.
3. Don't change rows, injection volume or # of injections:
    - Rows before samples:
        - Purge Injector; Condition Columns; Purge Detector; Equilibrate
    - Row after samples:
        - Flow down to 0mL
    - Injection volume: 40 µL and # of injections: 1.
4. Use "Run and Process". Click on the **GREEN ICON**. Click on Run.

RUN SAMPLE when instrument idle (only green icon is available):

1. Delete unused rows. Insert more rows if necessary- change name, vial #. Load carousel with your vials.
2. Don't change rows, injection volume or # of injections:
    - Rows before samples:
        - Condition Columns; Purge Injector; Purge Detector; Equilibrate
    - Row after samples:
        - Flow down to 0mL
    - Injection volume: 40 µL and # of injections: 1.
3. Use "Run and Process". Click on the **GREEN ICON**. Click on Run.

ANALYSIS:

1. **To Calculate MOLECULAR WEIGHT**: From the Empower Pro Window, open Browse Project → GPC DMF → ok. Open your run by double clicking left mouse button. Clear all integration using Edit→ Clear integration. Using left mouse button, integrate signal along the baseline and Quantitate (icon or Process→ Quantitate). Click on the icon Save ALL- close analysis window. Update Results. If you need to integrate few signals which are close to each another use CTRL-ALT and click with left mouse between signals
2. **To Create PDF REPORT**: With right mouse click on your updated analysis- **Preview/Publisher** –Open Preview/Publisher with left mouse- Use the Report Method- GPC Default Individual Report- OK. To change the scale in the report, close the first report window with right mouse click on the GPC results- Chromatogram Properties- Scaling- change Y-start and Y-end- Apply- close. You must save properties to be able to print. After you finish, please ALWAYS change properties- Y should be ~ 50 and save. Don't save any changes to the **GPC Default Individual Report**.

3. **To Change CALIBRATION (i.e. PMMA or PEG)**: To reprocess your open file with another calibration method, Open File→ Processing Method→PMMA cal. (latest date)- Open. Go to Edit→Clear Integration. Reintegrate with left mouse- Quantitate (icon)- Save All (icon)- Close Window- Update Results Table. Open results (Processing Method should be PMMA cal)- Print Report (see analysis).
4. **OVERLAY**: Empower- Results- with left mouse (SHIFT) select data- Tools- Compare.

UV DATA:

1. To view UV data, click on the channels tab in the Polymers-Results folder. Open the 2998 channel file for your sample.
2. To change wavelength- in the RUN window- Edit- UV- change wavelength (can be used from 200-400nm)- save. After you finish please change it back from 300-600 nm- Save.
3. Analysis- Channels- UV Data. Click on signal with right mouse- Extract Spectrum- Extract Chromatogram. Export UV data- copy and paste into Paint. Export data as Paint. 3D plot- Window- 3 D Plot

EXPORT DATA:

1. Empower- Browse Project- results-choose your run- Database- Export Method- ASCII- Browse- Choose Z: drive- write name of your file- OK
2. You will get 4 files in your folder if you choose export with PDF
    - .arw file is your raw data. This can be imported into Excel to graph
    - .ars is the software file
    - .pdf is the PDF of your data
    - .txt is the table that contains your Mw and PDI data after processing