GPC MALS – Manual

Polymer Facilities - MRL @ UCSB

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Sample Preparation. To be able to get absolute MW with dn/dc calculation - concentration of the polymer must be perfectly known. Sample must by clean, dry, and you need to prepare solution with exact concentration (3-4 mg/mL). Prepare solution with THF. As always, make sure your sample is completely dissolved, so you will not filter off part of the sample during filtration. Filter through 0.45 µm filter, and transfer sample into the autosampler vial (fill between the second and third mark).

- Solvent - THF
- Columns: Two Agilent PLgel MiniMixed-D bed columns + guard (MW linear range: 200-400,000 g/mol)

Use FBS to log into MALS computer & GPC MALS User account on the MALS computer

Loading samples on the Waters 2695 pump Using Empower:

1. STARTING EMPOWER SOFTWARE
    a. Open Empower icon on desktop (if you need to log in, the log in information is in the folder next to the computer).
    b. Click on Run Samples
    c. Open GPC MALS → GPC MALS. Click on Ok.
    d. Load previously created sample set (MALS mini no UV)
2. Delete unused rows. Insert more rows if necessary- change name, vial #. Load samples into autosampler carousel, and double check vial # in software with position in autosampler carousel.
3. Don't change rows, injection volume or # of injections:
    i. Rows before samples: Purge Injector; Equilibrate
    ii. Row after samples: Condition Column
    a. Injection volume: 40 µL and # of injections: 1.
4. Save, but **do not hit the run (Green Icon) until the Astra software is started!!!!** Note: You may have to enter a date after the Sample Set name (MALS mini no UV) to be able to save the sequence.

Entering samples Using Astra:

1. Open Astra from toolbar (blue icon)
2. Check instrument status by System → Instruments.
    a. A pop-up window will open. You should see 4 instruments present with green dots.
    b. You may need to click refresh in the pop-up window. If you do not see 4 instruments with green dots, you may need to restart the computer.
3. Create Astra sequence by File→ New→ Sequence from Empower
    a. Log into Empower
    b. Import Empower Sequence
    c. Open GPC MALS folder → MALS mini no UV
4. Go to Configuration, Samples and Collection Window:
    - Configuration- Click on the … → User → GPC MALS mini no UV
        - Click Apply at bottom of page
5. Samples- Insert sample concentration and dn/dc if known or 0.185 -- for PS (you can change value and recalculate for the correct value of dn/dc later).
    - Do not change run time = 35 min or injection = 40 µL.
6. Start run with Astra - using green RUN button.
7. Sequence will validate, and you will be able to save your sequence in your folder. After saving, you should see in the Collection window - Waiting for injection.
8. Press start (Green button) in the Empower software
9. Astra will automatically start data collection after sample injection.

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RESULTS: Analysis - using ASTRA.

1. Open ASTRA.
2. Open File→ Experiment→Sample name.
3. Open Procedures.
    a. Open Baselines
        i. Autofind baselines and adjust if necessary→ set all
        ii. Check baseline for each detector by clicking on the checkbox next to the detector number or letter code, adjust if necessary→ APPLY
        iii. When done with baselines→ OK.
    b. Open Peaks- Autofind peaks, can adjust peak window as necessary (To zoom in on baseline, use Ctrl + mouse left click)→APPLY→OK
4. Open Results→ Report (summary) to see Mn, Mw, Mp, PDI, etc.
5. Save

Dn/dc value – on line.

NOTE: You must assume 100% injection for calculation of dn/dc.

1. Open Astra.
2. Open File→ Experiment→Sample name, unless already open. Right mouse click on experiment→Apply Methods→ System→Methods→ RI Measurements→ 100% Mass Recovery Methods→ dn/dc from peak (should open new experiment file on the left side of Astra.
3. Open Procedures.
    a. Open Baselines, and set baselines if have not done so already
        i. Autofind baselines and adjust if necessary→ set all
        ii. Check baseline for each detector by clicking on the checkbox next to the detector number or letter code, adjust if necessary→ APPLY
        iii. When done with baselines→ OK.
    b. Open Peaks
        i. Autofind peaks, can adjust peak window as necessary (To zoom in on baseline, use Ctrl + mouse left click)
        ii. Check concentration of injected mass→APPLY→OK
4. Open Results→ Report (summary) New dn/dc value is under peak results
5. Enter new dn/dc value in Source Experiment file→Procedures→Peaks, type in dn/dc value
6. Open Results of Source Experiment File→Report (summary) to see recalculated Mn, Mw, MP
7. For completely new polymers or difficult systems it is possible to calculate dn/dc for 5 perfect concentrations of the polymer (for same solvent, temperature and wavelength of the laser)

Shutting down:

1. Check that system changes from waste to recycle mode.
2. Turn off laser on Heleos if not done by software