Preparative GPC Manual Krystyna Brzezinska (<u>kbrzez@mrl.ucsb.edu</u>) May. 2008

<u>SAMPLE PREPARATION</u>: Polymer 0.1 g or less dissolve into 2 mL of THF. Filter through 0.45 μ m filter.

OPEN PROGRAM: Empower- Run Samples- Water Diagnostic- Prep. GPC

AUTOMATIC INJECTION:

- 1. Load Samples- Load using "previously created sample set method"- GPC Prep.- OK.
- 2. Sample name- injection volume (up to 2 mL).
- 3. Slowly increase flow (0 6 mL/min). Method- GPC Prep1. <u>Stop Flow</u> icon use ONLY in the emergency situation. Recycle THF for 15-30 min to equilibrate system. Purge RI detector.
- 4. Wash injector few times with THF in the LOAD position. Load maximum 2 mL of the filtered solution into injector. USE SYRINGE WITH FLAT NEEDLE (Important!!).
- 5. Click on the GREEN icon- ok (a.) Sample Set- Setting up- b) Sample Set- Waiting for injection. Don't remove syringe. Move injector into INJECT position. Leave syringe until end of the run (45 min). Collect fractions.
- 6. After 45 min move injector into the LOAD position. Wash injector few times with THF. Injector is ready for next injection.
- 7. From time to time between runs inject pure THF to be sure that no polymer was left inside the injector or on the column.
- 8. <u>After last injection:</u> Move injector's arm into the LOAD position. Recycle THF moving tube into the pure THF bottle. Slowly decrease FLOW RATE from 6-0 mL/min. WASH injector with THF in the LOAD position.

MANUAL INJECTION:

OPEN PROGRAM: Empower- Run Samples- Water Diagnostic- Prep. GPC

MANUAL INJECTION (use if the automatic injection is broken):

- 1. Load Samples- Make single injection.
- 2. Slowly increase Flow (0 6 mL/min). <u>Stop Flow</u> icon use ONLY in emergency situation. Recycle THF for 15-30 min to equilibrate system. Purge RI detector.
- 3. Wash injector few times in the LOAD position with THF. Load maximum 2 mL of the filtered solution into injector. USE SYRINGE WITH FLAT NEEDLE (Important!!).
- 4. Click on **PREPARE** icon. Wait until **INJECT** icon on the desktop will be active.
- 5. INJECT sample moving injector's arm to inject position and same time click on **INJECT** icon on the desktop. Observe signal and collect fractions into the vials. After 45 min move injector's arm into the LOAD position and clean again injector few times with THF. You are ready to inject again.
- 6. From time to time between runs inject pure THF to be sure that no polymer was left inside the injector or on the column.
- 7. <u>After last injection</u> move injector's arm into the load position. Recycle THF moving tube into the pure THF bottle. Slowly decrease FLOW RATE from 6-0 mL/min. WASH injector in the LOAD position with THF.