

**GPC-DMF Manual**  
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**Sample preparation:** Prepare solution 4-6 mg of the polymer in 2 mL DMF with 0.1% of LiBr. Polymer **MUST BE SOLUBLE** in DMF. After filtration through 0.45  $\mu$ m filter transfer solution to the autosampler vials (3/4 of the vial must be filled) and load vials into carousel. System is using **DMF with 0.1% of LiBr** to prevent aggregation of the polymer.

Do not overload GPC columns. Sample concentration affect both viscosity and injection volume. While small sample amounts produce narrower peaks, viscous samples may require larger, more dilute samples. Table 1 lists the recommended concentration of sample for optimal results.

Table 1. Recommended Sample Concentrations:

Molecular-weight Range	Sample Concentration
0 to 25,000	<0.25%
25,000 to 200,000	<0.1%
200,000 to 2,000,000	<0.05%

**Add Sample to the running GPC-DMF:**

1. Edit- **Alter Running Sample**- OK
2. You should see two active icons: red and green. During run only red icon is active.
3. Insert rows, change name, vial # etc. After you finish changes click on the green icon and load carousels with your vials.
4. Don't change rows: Condition Column (ramp flow to 1 mL), Equilibrate (Method- Polymers) and Condition Column- Ramp Down Flow.

**Run Sample when instrument doesn't work or the Empower is closed:**

1. Open Empower:  
User Name and Password you will obtain after training. Click on Run Samples- Project- Polymers- Waters GPC2-OK
2. Load samples (icon or from File)- Load a previously created sample set method-choose Unknowns Sample Set- Open.
3. Insert more rows if necessary- Change vial #, Sample name. Use method "Polymers". Inject Broad Samples, Inj. Vol. 100  $\mu$ L and # of inj. 1. For automatic results use "Run and Report", continue on Fault- Run (the green icon).
4. Sample set- you can write your name or date- hit Run (the green icon) again.
5. You will find results on the printer. If you are not satisfied with integration you can change it (see analysis) and print it again.

**Analysis:**

1. Open "Browse Project" from the Empower Pro- Polymers.
2. Channels- RI detector.
3. With left mouse click twice on your results.
4. File- Open- Method Set- Polymers- Open.
5. Using left mouse integrate signal, Quantitate (icon), table-  $M_n$ ,  $M_w$  and PDI.
6. Save: File- Save- All or icon- close analysis window.
7. Open Results window from the Browse Project- Update.
8. Click on you analysis-With right mouse- Preview/Publisher -Open Preview/Publisher with left mouse- Use the Report Method- GPC Default Individual Report in the acquisition Method Set Polymers- OK.
9. Scale change: after closing report you will see second window. With right mouse click on the GPC results- Chromatogram Properties- Scalling- change Y-start and Y-end- Apply- close.
10. Print from File or printer icon.
11. To be able to print after scale was changed you have to save changes to GPC Default Individual Report. Click OK.
12. After printing go back to Y-start and Y-end values and change it back- save.