Chem/MatS/ChEn 4223W

Assignment 7

Due: In Lab, Tuesday, February 19/Thursday, February 21

This week you will be characterizing the molecular weights and weight distributions of the polymers you synthesized last week. You will be using gel permeation chromatography (GPC) to do this. In preparation for this lab, you may want to read:

Sperling, L. H. *Introduction to Physical Polymer Science* (Wiley-Interscience: New York, 2001), pp. 103-112.

Sandler, S. R.; Karo, W.; Bonesteel, J.; Pearce, E. M. *Polymer Synthesis and Characterization: A Laboratory Manual* (Academic Press: San Diego, 1998), pp. 140-146.

GPC is a relative (rather than absolute) method for determining molecular weight, and the instrument and column must be calibrated with a set of polymer standards. The set of 5 polystyrene standards you will use in this lab come from Agilent (<u>http://tinyurl.com/agilentgpc2</u>; EasyCal PS-2, *M*_w range 580-400,000). Data on this set of standards:

GPC CHROMATOGRAMS PS-2

KEY (Mp)	
1. 377,400	6. 188,700
2.96,000	7. 46,500
3. 19,720	8. 9,920
4. 4,490	9. 2,360
5. 1,180	10. 580

Columns: 2xPLgel 5µm MIXED-D, 300x7.5mm Eluent: THF Flow Rate: 1.0ml/min Detector: UV, 254nm



In the spectra above, the *x*-axis represents retention time. Notice that, characteristically for GPC, the highest masses elute first. The values listed represent M_x at each peak maximum. We will probably only use one of the two calibration sets (1-5 or 6-10); make sure you find out from the TA's which set we use.

The GPC column we will use is a Polymer Labs PLgel 10- μ m-pore MIXED-B column; some data on this column is available at <u>http://tinyurl.com/agilentgpc2</u>. What types of GPC columns are available? Name one, and describe what other sorts of polymers this column would have been more appropriate for.

Let's say our column became plugged with debris—sadly, something that happens once in a while when users don't filter their samples well—and we needed to purchase a replacement. But the folks at Polymer Labs and Agilent won't take our calls. Find an alternative column from a different manufacturer that we could use as our GPC column. I expect that the polymers you'll be making are between 10^4 and 10^5 g/mol; make sure your column could be used for that molecular weight range.

To collect your GPC data for this experiment, one of your group members will need to bring a USB flash drive.