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Molar Mass Analysis of Polymers

Objective:

The objective of this lab is to become familiar with two techniques for the analysis of molar mass in high-molecular weight materials. Two techniques will be used, gel permeation chromatography (GPC) {or high pressure liquid chromatography (HPLC)} and intrinsic viscosity using the Mark-Houwink equation. The samples are polystyrene.

Instruments to be used:

GPC (Crosley Hall, Prof. Neil Ayres (Leanne will help us)

Dilute solution viscometry (Departmental)

Materials:

Several polystyrene monodisperse standards and a commercial sample of polystyrene. Toluene (spectroscopic grade).

Procedure:

- 1) Prepare 1% by mass samples for the GPC in toluene.
- 2) Observed the operation of the GPC.
- 3) Determine the Mark Houwink parameters for polystyrene.
- 4) Prepare a series of solutions for determination of the intrinsic viscosity. Use the Ubbelohde viscometer to determine elution time for solvent and a series of polymer solutions.

Analysis:

- 1) Determine the molar mass for the samples studied using the Mark-Houwink equation.
- 2) Determine the number, weight and z-average molecular weights from the GPC curves.

Questions:

- 1) What moment of molar mass is obtained in intrinsic viscosity measurements? Is this an absolute or a secondary method? Is GPC an absolute or secondary method?
- 3) Is toluene a theta solvent for PS at the temperatures used? How do you know this?
- 4) How do the molar mass moments from GPC compare with the moment obtained from intrinsic viscosity?
- 5) From your experience in this lab, what are the advantages and disadvantages of GPC and intrinsic viscosity in the determination of molar mass?
- 6) If a GPC has only an index of refraction detector then how can the retention time be converted to molecular weight?
- 7) The GPC we used has a light scattering detector, an index detector and a viscosity detector. Explain how these can be used to obtain an absolute molecular weight from this instrument.
- 8) Light scattering usually involves the construction of a Zimm plot. Explain what a Zimm plot is and why a Zimm plot is this not necessary in the GPC? Under what condition is $M_w = M_n$?

1/21/00

More Specific Instructions for Capillary Viscometer:

1. Clean viscometer with toluene by rinsing at least 3 times, make sure the capillary has been rinsed.
2. Run pure filtered toluene (3 runs with the same time resulting). About 60 sec.
3. Make Initial Solution:
For 900 kg/mole PS 0.04g PS in a 25 ml Volumetric of Toluene.
For 100kg/mole PS 0.2 g/25ml toluene.
4. Drain the viscometer and add 9ml filtered solution.
5. Obtain 3 runs with identical time.
6. Add 6 ml filtered toluene, mix and run again.
7. Add 6 more ml filtered toluene, mix, drain to the line on left, and run again.