WHEN IS GPC AN APPROPRIATE CHARACTERIZATION METHOD?

WHEN IS GPC* AN APPROPRIATE CHARACTERIZATION METHOD?

Goal of GPC:

Obtain complete molecular weight distribution (MWD)

With multiple detector also obtain;

viscosity vs. mass

size vs. mass

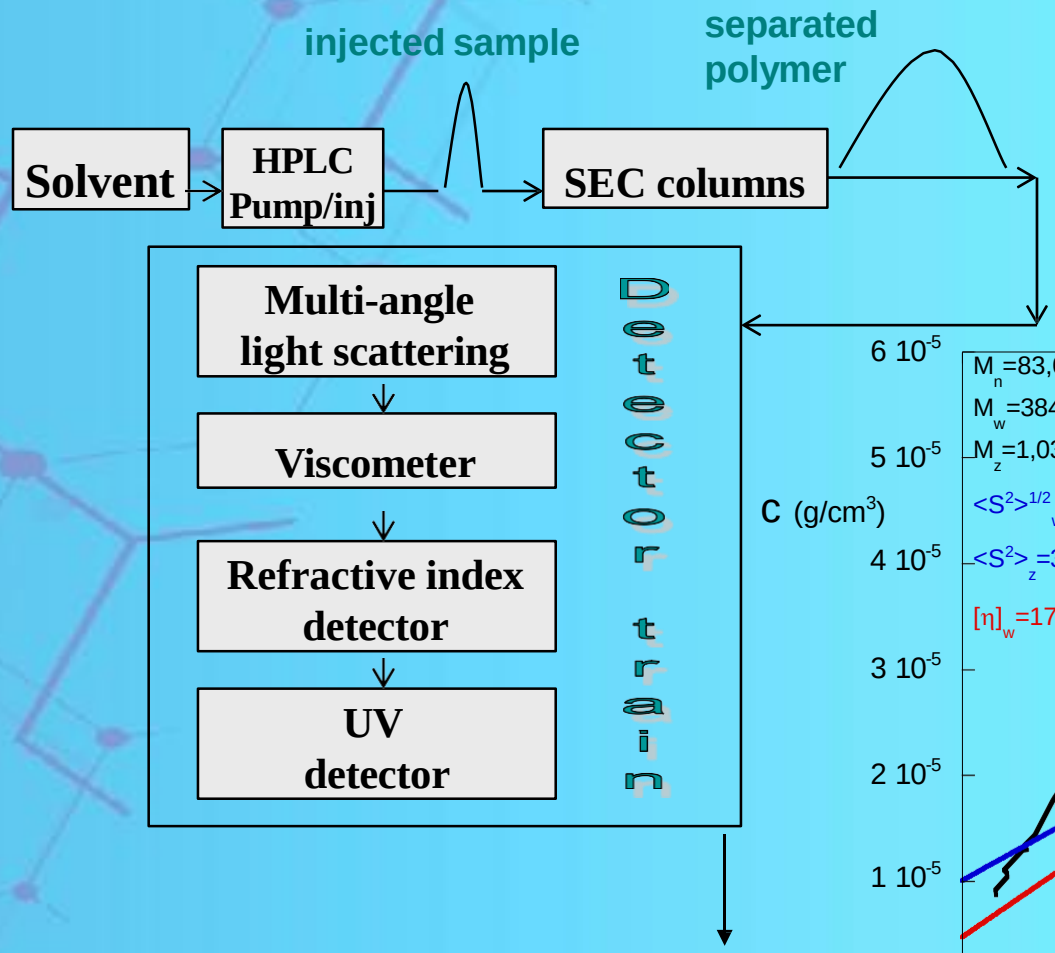
polyelectrolyte properties

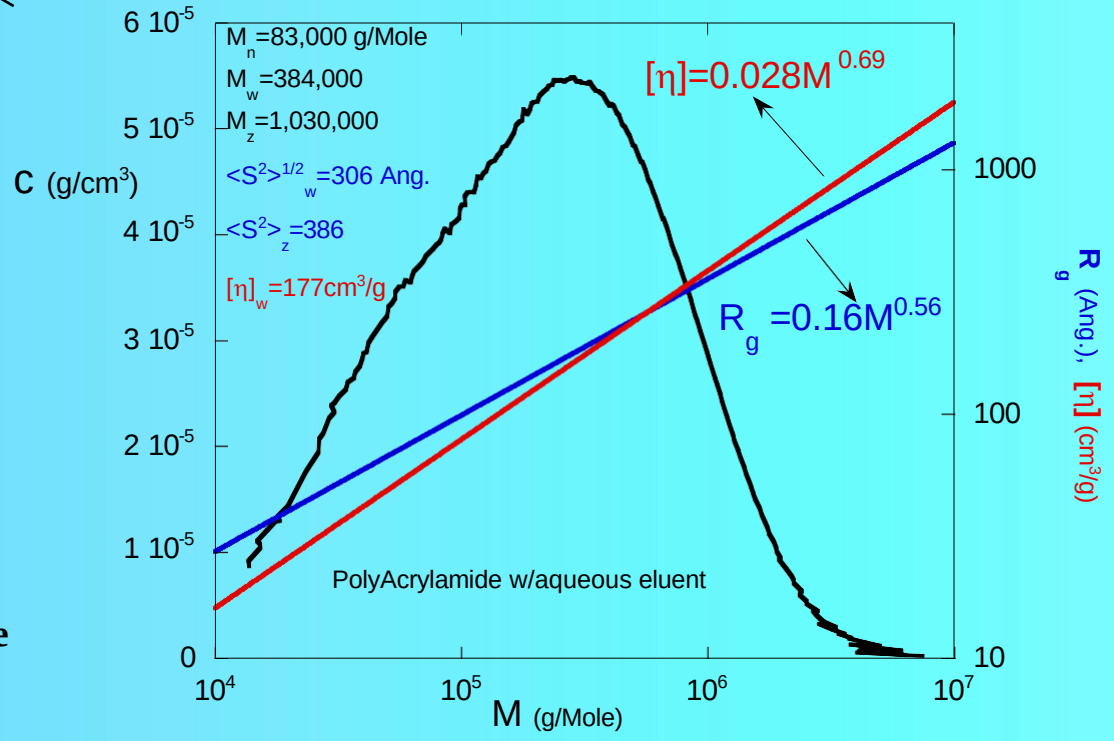
branching information

* Gel Permeation Chromatography (GPC) is also termed Size Exclusion Chromatography (SEC)

WHEN IS GPC AN APPROPRIATE CHARACTERIZATION METHOD?

Typical multi-detector GPC set-up and example of a characterized polymer





WHEN IS GPC AN APPROPRIATE CHARACTERIZATION METHOD?

GPC is meant for characterization of polymer solutions *in Equilibrium*

• Many polymer solutions are ***unstable***. Solutions with a strong propensity for instability through aggregation and phase separation include those containing:

Proteins

Polysaccharides

Other natural products

Polymers in poor solvents

Copolymers and complex polymers (e.g. branched/cross-linked)

• When characterizing new polymers and/or new solvents, the solutions should be tested for stability.

• Instability may occur over periods of seconds, hours, days, or months.

WHEN IS GPC AN APPROPRIATE CHARACTERIZATION METHOD?

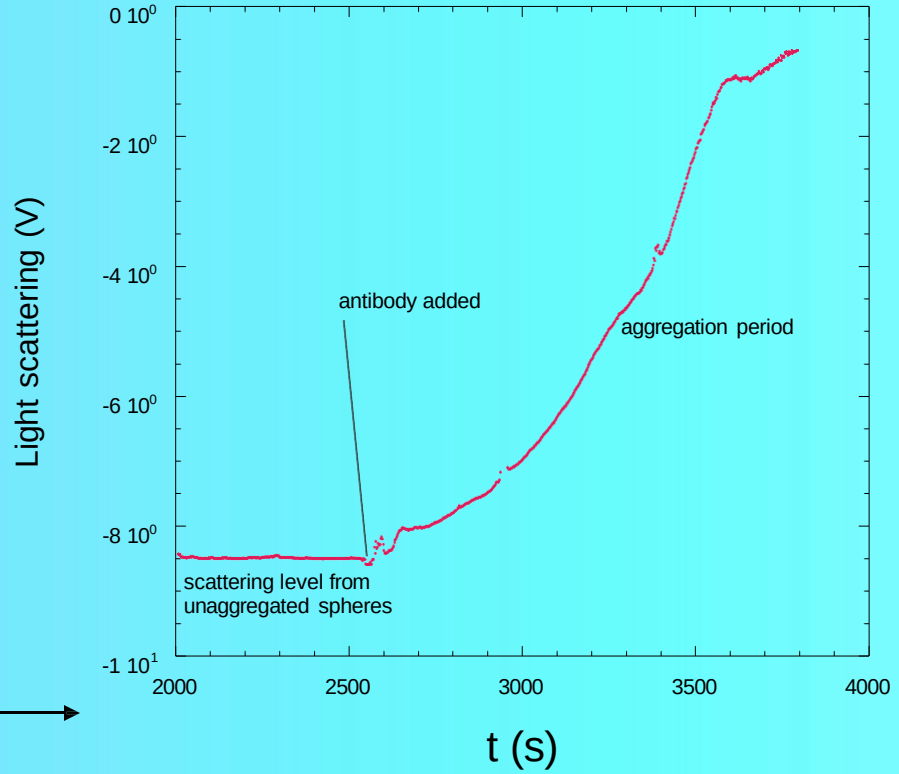
Means of assessing polymer solution stability, ***before starting GPC studies***

- Light scattering is exquisitely sensitive to even small amounts of aggregation; e.g. typically, it is possible to detect one dimerization event among a hundred protein molecules

- Aggregation in solution may produce no visible turbidity at all, but light scattering will immediately detect aggregation events.

- ***Batch*** light scattering is well suited for detecting time-dependent aggregation.

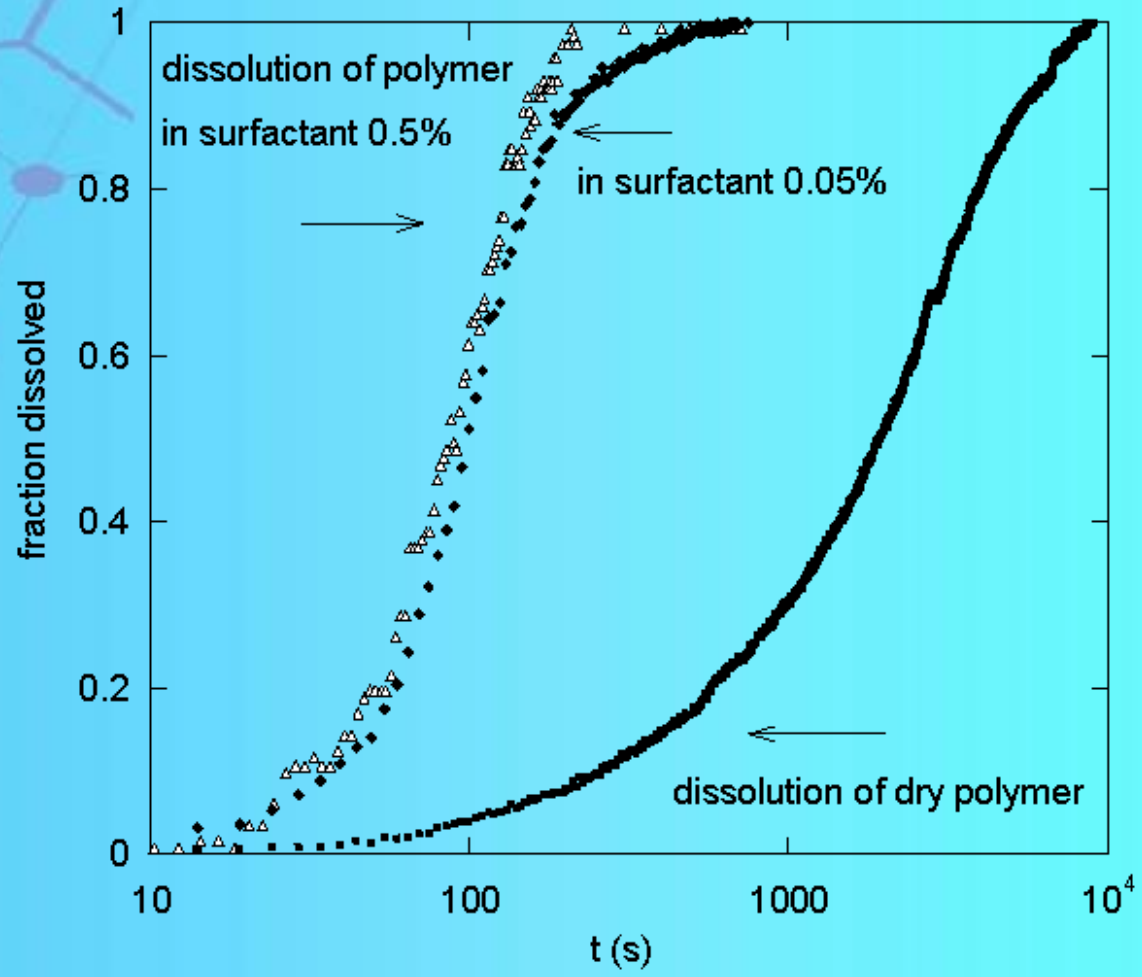
E.g. Immediate detection of aggregation of protein coated 5nm gold nanospheres when antibody is added. It takes many hours to develop any measurable turbidity.



When is GPC an appropriate characterization method?

Polymer should be *fully dissolved* before GPC injection

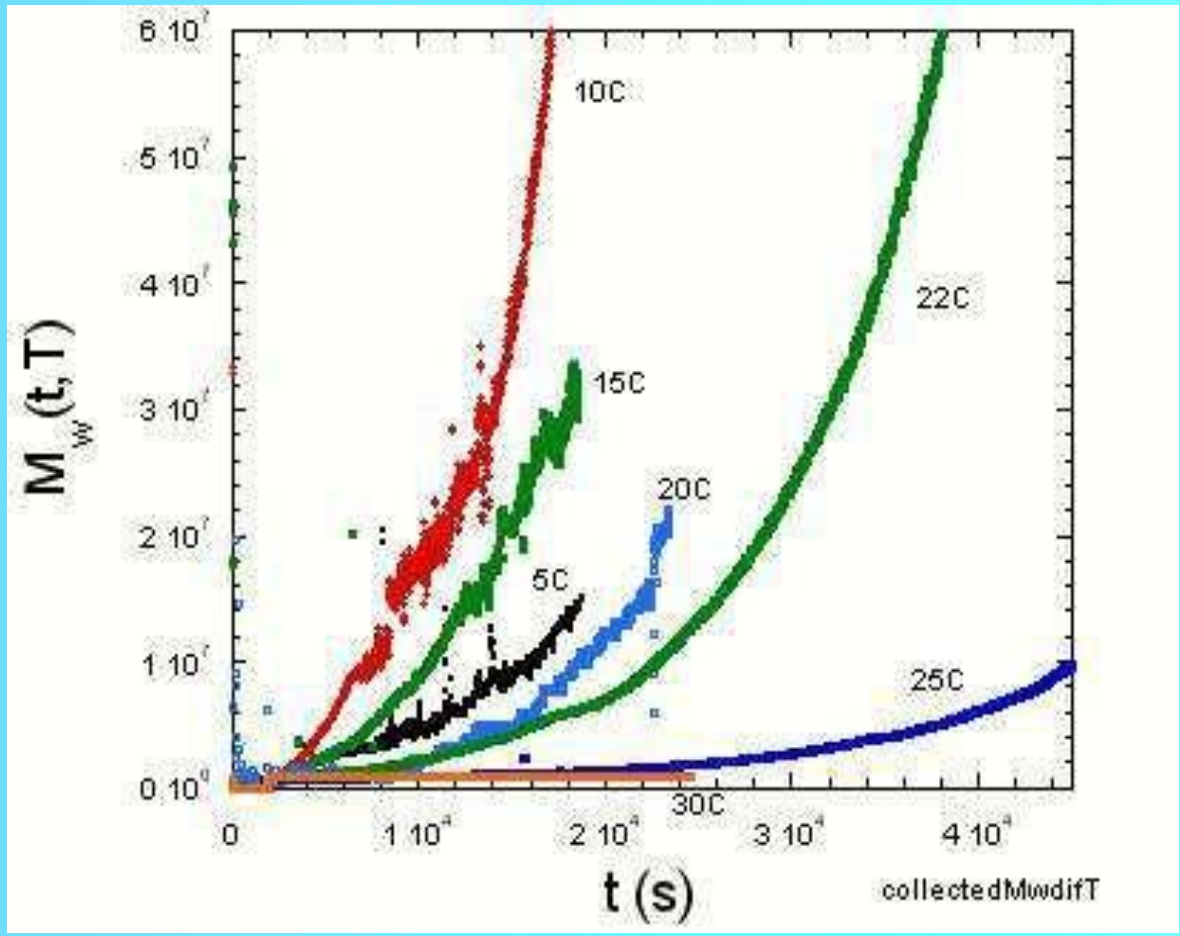
Dissolution kinetics of polymer in inverse emulsion, and from dry state



WHEN IS GPC AN APPROPRIATE CHARACTERIZATION METHOD?

An example of a *non-equilibrium* polymer solution: gelatin aggregation at different temperatures

Aggregation process for 0.75% gelatin solutions at different temperatures



WHEN IS GPC AN APPROPRIATE CHARACTERIZATION METHOD?

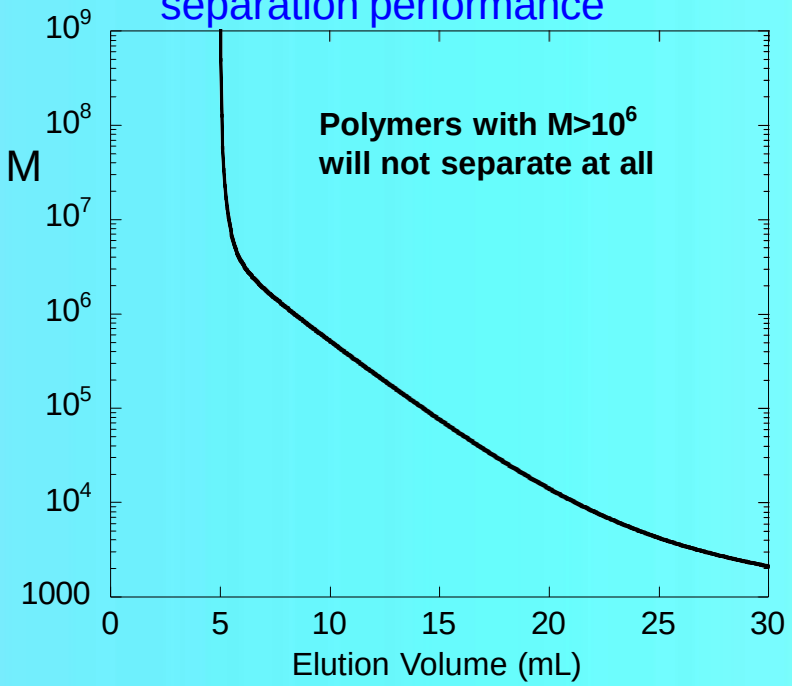
Besides instability of solutions, there are other cases where GPC may not be appropriate

- Polymers *too large for separation on the GPC column* simply elute in the exclusion volume; i.e. one essentially performs a batch measurement.

- This will give an artificially low polydispersity.

- Large polymers are often shear sensitive and can be degraded going through columns.

A generic column separation performance



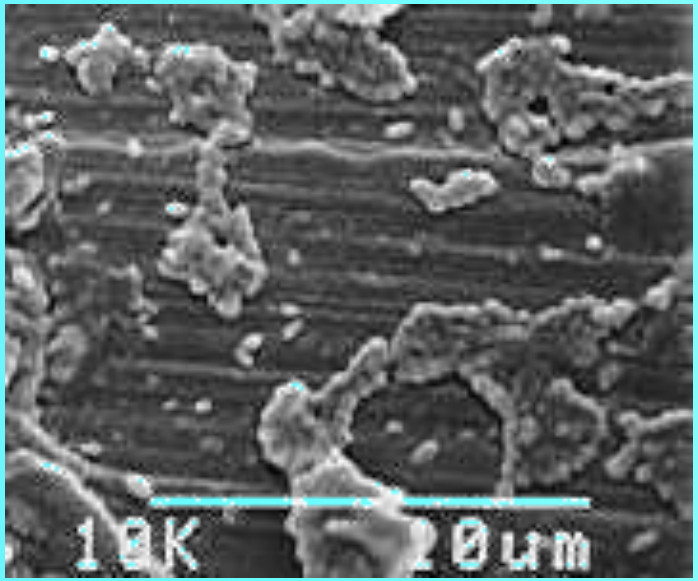
WHEN IS GPC AN APPROPRIATE CHARACTERIZATION METHOD?

- Besides instability of solutions, there are other cases where GPC may not be appropriate

- Solutions containing *microgels* that will be trapped in the GPC column; not only is the character inaccurate, *expensive columns can be damaged*.

- Polymers that contain other large particles; e.g. microcrystals sometimes form during polymerizatio these can destroy columns like 'cannon balls' shooting through.

- *SEM of extremely hard microcrystals produced in a fluoropolymer emulsion polymerization; the small spheres surviving filtration destroyed GPC columns*

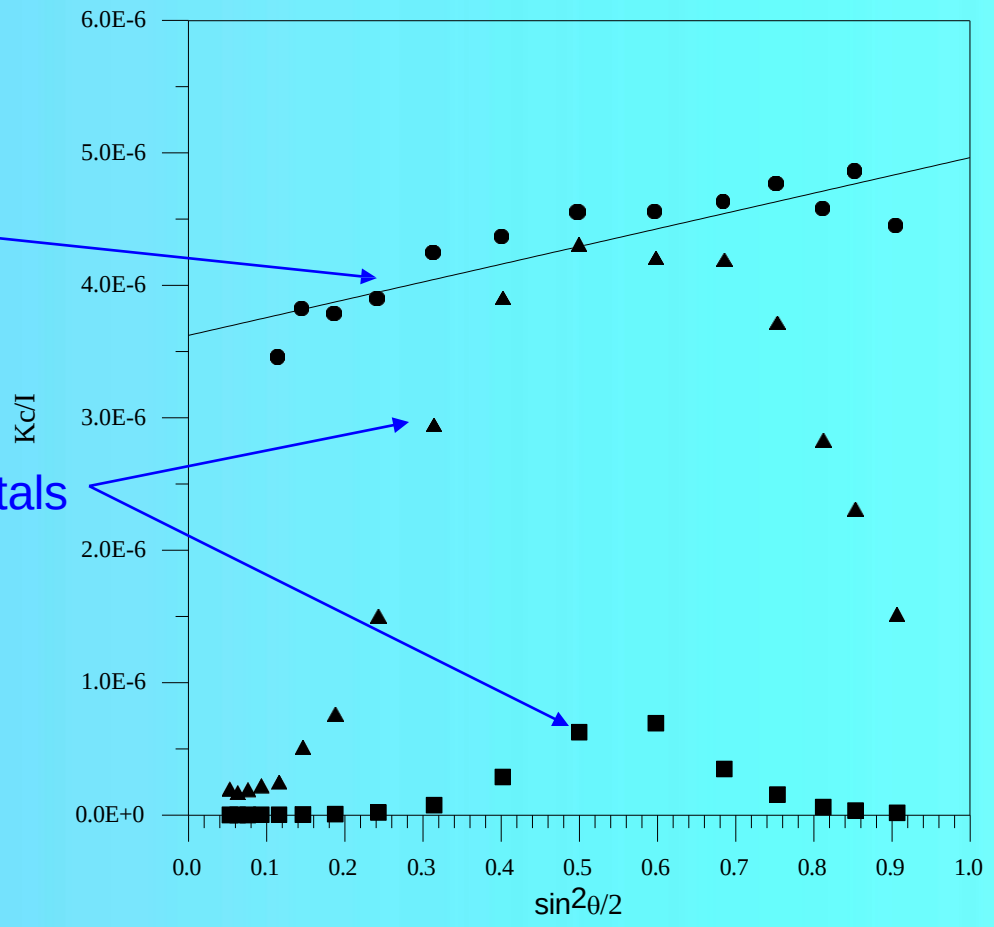


WHEN IS GPC AN APPROPRIATE CHARACTERIZATION METHOD?

Detection of microcrystals which can destroy GPC columns using ***Batch*** light scattering, ***prior*** to GPC

Fluorinated polymer without Microcrystals (removed by centrifugation)

Fluorinated polymer with microcrystals



ONCE IT HAS BEEN DETERMINED THAT THE POLYMER/SOLVENT SYSTEM IS APPROPRIATE FOR GPC…..

Let the GPC begin….