Application Notes

Aramide—Thermal FFF

olyesters and polyamides are notoriously difficult to characterize using traditional techniques such as GPC. They are hard to dissolve and may contain high molar mass constituents or microgels—which may themselves be important to the final properties of the materials. In addition, even when the materials can be solubilized in NMP for GPC, they require salts to suppress interactions of the polymer with the column matrix. Finally, even when samples *can* be prepared adequately, traditional GPC offers no hope of characterization, since no appropriate calibration standards exist.

A superior method of characterization was performed by the Hoechst AG (Frankfurt, Germany) Central Research facility, wherein a T-100 thermal fractionator (FFFractionation, Salt Lake City, UT) was used to perform the separation, and a multi-angle light scattering instrument (DAWN DSP) for the absolute molar mass and size determinations. The DAWN DSP used here had an argon-ion laser for maximum sensitivity at low concentrations.

Using a UV detector in series with the DAWN, absolute molecular weights were determined; and the DAWN's unique multi-angle detectors allowed the root mean square size to be determined as well. No calibration standards were used, since the combination of the DAWN and UV signals create absolute molar masses. As no salts were needed in the mobile phase the molar mass values are determined unambiguously. The specific refractive index increment, dn/dc, was measured off line in NMP.

The two graphs on the right illustrate vividly how efficiently the separation was achieved by this novel thermal FFF technique.

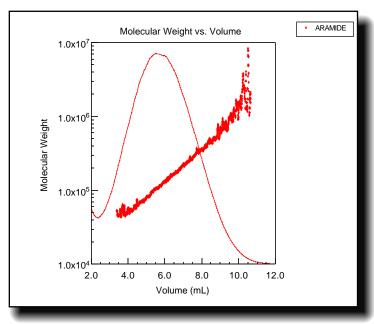


Figure 1. Beautiful separation has been achieved for a sample that is virtually impossible to handle with conventional GPC.

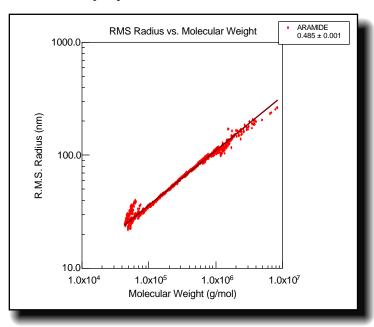


Figure 2. The conformation plot can be used to determine the extent to which the polymer is uncoiled—or unbound. This is seen in the slope value reported in the legend (upper right).

