Waters
Lab Highlights
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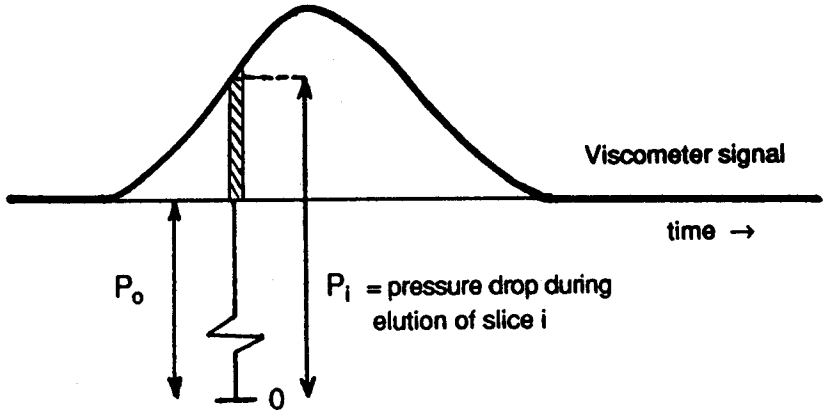
The 150CV GPC/Viscometer System IV. Viscometer and RI Detector Responses

This is the fourth in a series[1-3] of Lab Highlights which describe the operation and performance of the 150CV GPC/Viscometer System. This Lab Highlight describes the basic response characteristics of the single-capillary viscometer detector and the differential refractive index (RI) detector in the 150CV system. Each detector responds to eluting sample in a different way and the combination of both detectors is a powerful tool for polymer characterization.

Viscometer Detector

The viscometer generates a signal equal to the pressure drop across the small viscometer capillary[3]. When no sample is eluting from the columns, the viscometer measures the pressure drop (P_0) of the pure solvent flowing through the capillary. This background pressure is typically at 40 to 70% of the full scale range of the differential pressure transducer (5 KPA; ~0.73 psi). For example, in THF (35°C) at a flow rate of 1 ml/min, P_0 is ~2.5 KPA with the standard capillary (6" x 0.014" I.D.). The polymer signal is a very small change in this background signal. During elution of the polymer, the maximum pressure drop increases only ~1% above the value of P_0.



A computer data system can easily plot the response of the viscometer detector by subtracting most of the baseline signal. In order to plot this polymer signal on a standard chart recorder to monitor detector and system performance, the viscometer electronics controls on the 150CV front panel are used to remove the background signal (P_0) so that the small, incremental polymer signal can be expanded ~50 to 100x to properly display the chromatogram. The 5 volt output signal of the viscometer transducer is passed through an auto zero circuit when no sample is eluting to offset the background pressure drop (P_0) and the incremental signal due to the sample is displayed on a 50 mv chart recorder (100x amplification) to plot the chromatographic output of the viscometer detector.

Juris Ekmanis

Waters
Division of MILLIPORE 34 Maple Street Milford, MA 01757 508-478-2000

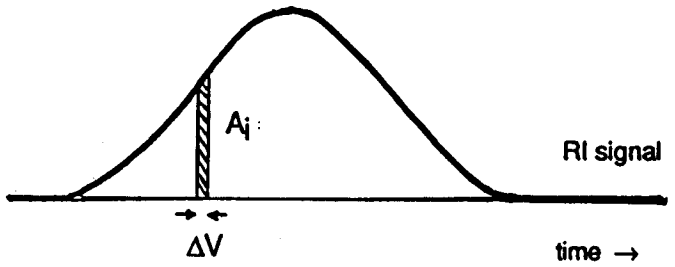
Using the relationship for reduced viscosity, the intrinsic viscosity of column effluent at any slice (i) in the viscometer signal can be calculated as follows.

$$[\eta_i] = (P_i - P_o) / (C_i \cdot P_o)$$

where: $[\eta_i]$ = intrinsic viscosity of slice i
P_i = total viscometer signal at slice i
P_o = viscometer background signal at slice i
C_i = concentration of sample in slice i

Refractive Index Detector

The differential refractive index detector is a concentration detector and affords a signal directly proportional to the concentration of polymer in the column effluent. For a given polymer/solvent system, the RI response is independent of the molecular weight of the polymer for molecular weights above ~2,000.



The concentration of sample in the column effluent at any slice in the polymer region of the RI response can be calculated using the RI data as follows:

$$C_i = (C \cdot V \cdot A_i) / (DV \cdot A)$$

where: C_i = concentration of sample in slice i
C = concentration of polymer in solution injected
A_i = area of slice i
A = total area of RI response for polymer sample
V = injection volume
ΔV = volume of slice i

This relationship requires that the RI response represents the total mass of sample. If any portion of the sample (e.g. insoluble fillers or crosslinked material) is not soluble in the solvent, the value for polymer concentration (C) must be corrected to indicate the true concentration of the dissolved polymer which elutes under the polymer envelope on the RI detector.

References

1. LAH 0436 6/90.
2. LAH 0437 6/90.
3. LAH 0441 11/90.