CHROM. 5137

THE EFFECT OF TEMPERATURE ON GEL PERMEATION CHROMATOGRAPHIC SEPARATIONS

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SUMMARY

In three previous studies[1–3] we reported the effect of several operational parameters on separations by gel permeation chromatography. These studies included the effects of (a) solvent flow rate, (b) sample concentration, (c) sample molecular weight, (d) particle size, and (e) operating temperature. In this present study, we extended the operating temperature limit to temperatures above the normal boiling point of the solvent. At these temperatures, polymer solutes will have smaller diffusion coefficients (solute moves faster) and the solvent will be less viscous. Both of these effects enhance the mass transfer of the solute and lead to more efficient separations.

A modified Waters Associates gel permeation chromatograph, Model 200, was operated with toluene solvent. The instrument was modified to include pre-columns for heating the solvent and a restrictor and cooling bath (before the inner heat exchanger of the refractor) to keep the solvent in the liquid state. The restrictor is necessary when operating above the boiling point of the solvent.

Two 4 ft. × 0.305 in. I.D. columns containing 10^5 Å Styragel were used for all studies. A siphon calibration curve was used to correct inaccuracies in the siphon dump volume. Samples were injected at a concentration of 0.5 % (weight/volume). A constant sample volume of 1 ml was used for all test samples. Samples of polystyrene standards were evaluated at 32, 80, 135 and 200° and at flow rates ranging from 1 to 10 ml/min. Elution volumes were found to decrease with temperature since the solute molecules are more fully expanded and occupy a larger volume at the higher temperatures. Peak widths decreased with temperature due to the better mass transfer. The column system gave a plate count (ODCB) of 3000 plates/ft. when operating at 1 ml per min and 135°. By increasing the temperature from 32° to 135°, the flow rate could be increased six-fold and maintain constant resolution. Since the solvent viscosity is much lower, the increased temperature and flow rate only increases the pressure drop by a factor of three. The data for all the standards and conditions were compared on a resolution (R), time (t), R/t and pressure drop basis.

REFERENCES

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