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Characterization and Properties of Macromolecules. VIII. A Study of Experimental Variables in Preparative-Scale Gel Permeation Chromatography

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Synopsis

Three preparative-scale gel permeation chromatography columns were constructed and filled with Corning porous-glass packing materials. Each column was packed with a different pore-size material, CPG 10–2000, CPG 10–350, CPG 10–120, all of which had been treated with hexamethyldisilazane. An additional preparative-scale column packed with Styragel, nominal porosity 5×10^4 Å, was added to complete the column bank. Polystyrene standards were used to investigate the effects of molecular weight and sample concentration upon elution volume. A commercial polystyrene sample was fractionated using this system to study the effects of sample concentration and flow rate on fractionation efficiency. These fractions were analyzed by high-resolution analytical-scale gel permeation chromatography. Where possible, the results have been compared with similar studies that used Porasil (Spherosil) and Styragel columns.

INTRODUCTION

Although gel permeation chromatography (GPC) has been widely used for polymer characterization in recent years, relatively little work has been reported on large-scale separations by this technique in nonaqueous systems. Large-scale fractionation is required to provide samples of narrow molecular weight distribution (MWD) for subsequent mechanical testing, rheological studies, and other physical measurements.[1]

Naturally, any study of experimental variables in preparative-scale GPC will involve a choice of some efficiency parameter which measures column performance. There are many available choices, for example, the number of theoretical plates, n (defined later in the paper). This may be calculated for monodisperse polymeric species; but because these are unobtainable, this measurement is usually made with a low molecular weight solute. Other available appropriate parameters include the resolution R (defined later in the paper), which is a mathematical test to determine if two peaks are separated, and the resolution index RI (defined later in the paper), which has been proposed to account for the fact that the GPC calibration curve, molecular weight versus elution volume, is a logarithmic function.

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Usually, the purpose of a preparative-scale GPC fractionation is to produce narrow MWD fractions. Thus, the quality of the fractions, $\bar{M}_w/\bar{M}_n$, measured, for example, by analytical-scale GPC, produced under a given set of operating conditions will serve as a basis for comparison of column efficiency. We consider this to be a valid method to measure column efficiency for the purposes of this study, provided precautions are taken to correct the molecular weight averages from analytical GPC for band broadening (vide infra). The value of $\bar{M}_w/\bar{M}_n$ obtained from analytical-scale GPC has previously been used to measure column efficiency in preparative-scale GPC, but the results have often not been corrected for band broadening. Generally, previous workers have only used one of these efficiency parameters, making comparisons with the present results difficult.

Dark, Levangie, and Bombaugh[2] fractionated polyethylene using a single 4-ft × 2.5-in.-O.D. Styragel column with a nominal porosity of 10^5 Å. Fractions with values of $\bar{M}_w/\bar{M}_n$ from 1.23 to 1.44 were obtained, with no systematic variation of $\bar{M}_w/\bar{M}_n$ with molecular weight. Increasing sample size decreased fractionation efficiency, although a limit was found below which no further increase in efficiency with decreasing sample size was observed. Flow rate studies given in this reference for two Styragel columns 4 ft × 2.5 in. O.D. in series with porosities of 10^6 and 10^4 Å showed little decrease in efficiency with increasing flow rate. In a subsequent paper, Bombaugh, Dark and King[3] evaluated the effect of sample injection volume on column efficiency and noted that there is a limiting volume above which efficiency decreases. This result was obtained using the number of theoretical plates for a small molecule as a measure of column efficiency.

Law,[4] using a 4-ft × 2.0-in.-O.D. column packed with equal amounts of 250 and 1000 Å Styragel, fractionated carboxypolybutadienes and found $\bar{M}_w/\bar{M}_n$ values from 1.15 to 2.26. These values increased regularly with increasing molecular weight. Bombaugh and Levangie,[5] in further studies using one 4-ft × 2.5-in.-O.D. Styragel column of 2.5 × 10^4 Å porosity, showed results that indicate the maximum amount of polymer that can be injected for good resolution is about 1 g in a volume of no more than 100 ml for this column type and size. Montague and Peaker[6] describe a large-scale GPC apparatus. Their results show that the narrowest distribution fraction was that with the highest molecular weight.

Peyrouset and Panaris[7] studied preparatory-scale GPC using Porasil (Spherosil) packing materials. Polystyrene fractions with $\bar{M}_w/\bar{M}_n$ values from 1.10 to 1.19 with no obvious dependence on molecular weight were obtained. The column efficiency, in terms of plates per foot, were 750–875 for 2.36-in.-O.D. columns, about twice the values observed for $^3/_8$-in.-O.D. analytical columns packed with the same materials. Similar results showing that preparative columns are more efficient than analytical columns have been reported.[6]

This study is concerned with the effect of sample concentration on elution volume, sample concentration on efficiency, and flow rate on efficiency for preparatory-scale GPC columns using porous glass packings.

EXPERIMENTAL

Apparatus

A Waters Associates ANA-PREP gel permeation chromatograph was used. The pump was a series-connected duplex Milton-Roy Mini-Pump with a maximum flow rate of 140 ml/min. The injection loop volumes were either 50 ml or 100 ml; the solvent used was tetrahydrofuran (THF) containing 1 g butylated hydroxytoluene (BHT) per 5 gallons (US) of THF. From a consideration of the total liquid volume of the columns and the fact that the fraction collector has only 40 ports on the rotary valve, the fraction collection volume was set at 200 ml. This was the minimum volume which still allows the unit to be operated in the automatic mode. The elution of sample from the preparative columns was monitored using a differential refractometer.

Immediately after the column, the flow was split so that 1 ml/min was diverted through the detector. The fraction collector empties by the application of nitrogen pressure, which momentarily increases the flow rate through the splitter. This puts a spike on the chromatogram and serves to monitor elution volume.

Column Construction

The Styragel column was a commercially available column (Waters Associates, Milford, Massachusetts) packed with 5×10^4 Å material. The dimensions were length = 4 ft, O.D. = 2.5 in., I.D. = 2.25 in. The other three columns were packed with Corning porous glass (Electro-Nucleonics, Fairfield, New Jersey). These were treated with hexamethyldisilazane[8] to eliminate or reduce adsorption problems. The column packed with CPG 10–350 had identical dimensions to the Styragel column. The other two porous glasses, CPG 10-2000 and CPG 10-120, were packed in columns constructed from tubing having I.D. = 2.625 in. and length 33.5 in. in order that these columns should have identical internal volumes with the previous two columns. The end plates were attached, using six bolts, to a collar welded at each end of the column. A recess was machined into the end plate to accomodate a filter which protruded $^1/_{32}$ in. above the face. The filter material was porous stainless steel (Mott Metallurgical, Farmington, Conn.) $^1/_8$ in. thick and of 20-μ porosity. This type of column fitting has been shown[9] to provide a very uniform spreading of a sample onto a large-diameter column. A groove was machined into the collar to accommodate a Vanway O-ring which effected a liquid seal. The O-ring chosen was a Teflon-covered elastomer (Chesterton-Vanway, Stoneham, Massachusetts). These O-rings are superior to Teflon, i.e., they have the same solvent resistance but do not deform under pressure so that the column end can be removed and reinstalled many times using the same O-ring.

Order of Columns

Previous descriptions of preparative-scale gel permeation chromatography have not stated in which order the columns were installed. Intu-

itively, it seems that the small pore-size columns be placed first, followed by columns having successively larger pore sizes. This is the order in which the separation takes place, and hence there is no overtaking of one molecular weight species by another. Altgelt[10] has shown that unusual elution effects can occur in this situation, particularly with high loads. Ouano,[11] however, considers the ordering used here to be "reverse" to the "normal" (presumably that ordering which most chromatographers use, although this information is rarely provided). He suggested a random ordering would be the best method for highest resolution. However, it is impossible to arrange a random ordering with four columns; but this question of column ordering deserves further study, particularly for preparative scale GPC.

Analysis of Fractions

For each sample concentration studied, fractions were taken using the automatic fraction collector. Fractions, spaced at suitable intervals throughout the chromatogram, were selected for subsequent analysis by analytical-scale gel permeation chromatography. A planimeter was used with the preparative chromatogram to calculate the concentration of each fraction to be analyzed, and a suitable dilution was made. The analytical scale GPC was carried out using four columns, each 6 ft in length, having a 0.375-in. O.D. and a 0.305-in. I.D., and bent into a U shape. These columns were packed with Corning porous glass CPG 10-240, CPG 10-370, CPG 10-1250, and CPG 10-2000 which had been treated with hexamethyldisilazane. The flow rate of tetrahydrofuran was 1.5 ml/min, and flow through the columns was in the order in which they are listed above. The sample concentration was 1 mg/ml, and the injection volume was 2 ml. Column calibration was carried out with narrow molecular weight distribution polystyrene standards (Pressure Chemical Co., Pittsburgh, Pennsylvania).

RESULTS AND DISCUSSION

Initially, the preparative GPC flow rate was 50 ml/min which corresponds to approximately the same linear flow velocity as running an analytical GPC column having a 0.305-in. I.D. at 1 ml/min. The calibration curve for this column series arranged in the order previously given, run at 50 ml/min flow rate, is shown in Figure 1. Previous work had not reported if the order of columns arrangement or the concentration of injected polymer solution had an effect on elution volume in preparative-scale GPC. A previous study[12] using analytical GPC suggests that concentration effects would be important. As can be seen from Figure 1, increasing the sample concentration, at constant injection volume (50 ml), causes a given polymer to elute later. For example, using the 1 mg/ml calibration curve would lead to considerable errors in assigning molecular weights at other concentrations. When the 200,000 molecular weight

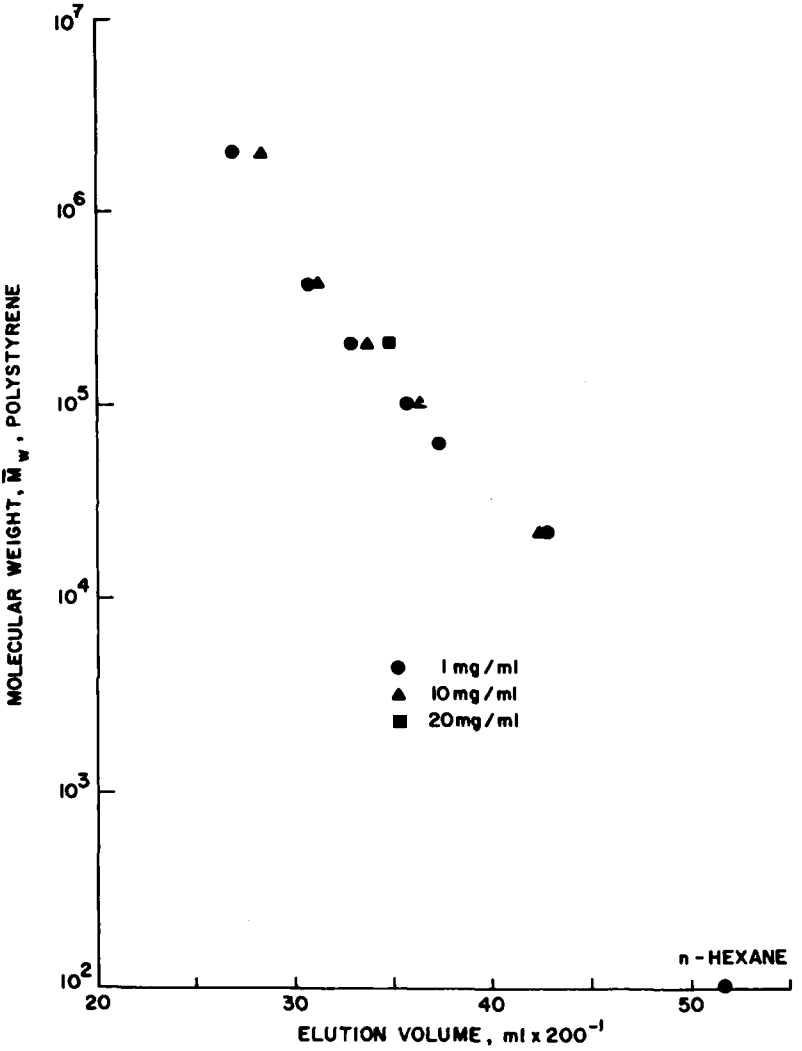

Fig. 1. Effect of molecular weight and sample concentration upon elution volume for preparative GPC columns: polystyrene solutes, tetrahydrofuran solvent, and 50 ml/min flow rate.

polystyrene is injected at 20 mg/ml concentration, the molecular weight appears to be 120,000 as determined by using the 1 mg/ml calibration curve. Thus, it is very important to ensure that the sample concentration is kept constant when performing preparative-scale GPC. An interpretation of this effect has been given, using the dependence of polymer coil size on concentration.[12]

A typical preparative scale chromatogram is shown in Figure 2 which shows the polystyrene standard with $\bar{M}_w$ = 97,200 run at 10 mg/ml. An analysis of this chromatogram by a computer program[13] using the calibra-

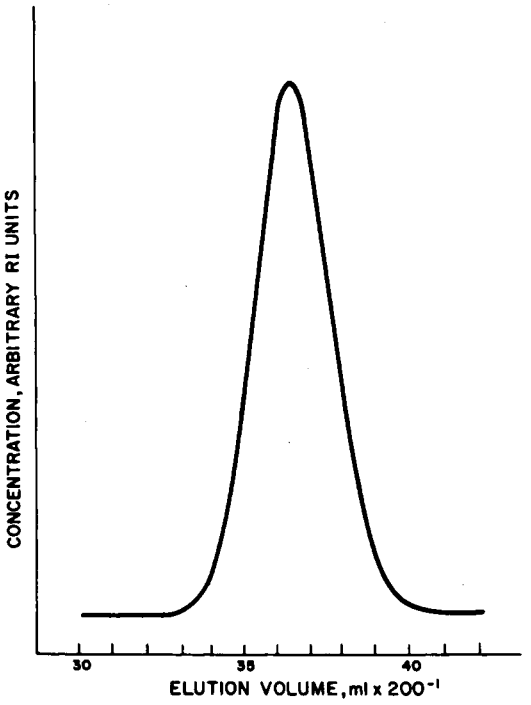

Fig. 2. Chromatogram of polystyrene standard, $\bar{M}_w$ = 97,200, obtained at flow rate of 50 ml/min with tetrahydrofuran solvent: sample concentration, 10 mg/ml; injection volume, 50 ml.

tion curve shown in Figure 1 at the appropriate concentration yielded the following: $\bar{M}_w/\bar{M}_n = 1.09$. This value indicates high column efficiency since the expected value assuming infinite resolution is 1.06. Table I lists values of the number of theoretical plates per foot, n, the resolution R, and resolution index RI, calculated from the formulae

$$n = \frac{1}{L}\left(\frac{V_e}{W_b/4}\right)^2$$

$$R = 2\,\frac{(V_{e1} - V_{e2})}{(W_{b1} + W_{b2})} \qquad V_{e1} > V_{e2}$$

$$RI = \left(\frac{\mathrm{MW}_1}{\mathrm{MW}_2}\right) 1/R \qquad \mathrm{MW}_1 < \mathrm{MW}_2.$$

where L is the column length (ft), V_e is the elution volume, and W_b is the peak base width.

At each concentration, the number of theoretical plates increases with decreasing molecular weight. The values of n for 1 mg/ml concentration are quite similar to those using analytical-size GPC columns at comparable flow velocities.[14] At 10 mg/ml concentration, the values of n are smaller than those obtained at 1 mg/ml concentration at each molecular weight.

TABLE I
Values of Number of Theoretical Plates, Resolution, and Resolution Index Calculated from Preparative Column Chromatograms

Molecular weight $\bar{M}_w$	Concentration, mg/ml	n, plates/ft	Polymer pair $\bar{M}_w$	Concentration, mg/ml	R	RI
			411,000 / 200,000	1	0.52	0.25
411,000	1	59	411,000 / 200,000	10	0.44	0.20
	10	40				
200,000	1	76	97,200 / 20,000	1	1.51	0.35
	10	53				
	20	42				
97,200	1	77	97,200 / 20,000	10	1.16	0.25
	10	66				
20,000	1	91				
	10	70				

This decrease in efficiency is also evident from the values of resolution R. For a given polymer pair, the value of R is lower at the higher concentration indicating poorer column efficiency. Similar conclusions can be drawn from the resolution index (RI) values.

In order to evaluate the effect of polymer concentration and molecular weight on column efficiency, a different series of experiments was conducted. For this purpose, commercial polystyrene was chosen with $\bar{M}_w/\bar{M}_n \sim 2$. This injection loop was changed to 100 ml in order to reduce the viscosity of the sample solution at a given weight of polymer. Using the ANA-PREP with this series of columns in automatic mode limits the minimum fraction volume to 200 ml. Thus, even with the 100-ml injection loop, the injection volume is still only 50% of the collection volume.

The commercial polystyrene was injected at the following concentrations (g/100 ml THF): 0.5, 1.0, 1.5, and 2.0. Fractions of 200 ml each were taken and analyzed by analytical GPC. A composite figure of the preparative chromatograms for the 0.5 g/100 ml and 2.0 g/ml injections is shown in Figure 3. Originally, it was intended to take five fractions for analysis at the same elution volumes for each of the four fractionations performed at the different concentrations. However, as is evident from Figure 3, the chromatogram is displaced to higher elution volumes at the higher concentration. In order to take fractions at approximately the same molecular weight in these experiments, the fraction number to be analyzed was varied and is indicated by the arrows. These fraction numbers are included in Table II and indicate the number of 200 ml fraction volumes eluted since the sample was injected.

In two cases, the flow rate was varied in order to reduce the separation time. At a concentration of 1.5 g/100 ml, the flow rate was increased to 80 ml/min and then to 140 ml/min. The chromatograms are shown in Figure 4, and the fractions collected for analysis are indicated by arrows.

TABLE II

Values of $\bar{M}_w$ and $\bar{M}_w/\bar{M}_n$, Uncorrected for Band Broadening, for Fractions Obtained from Preparative-Scale GPC Fractionation of Shell Crystal-Grade Polystyrene

Flow rate, ml/min	Injected concentration, g/100 ml							
50	0.5	Fraction number	29		34	37	40	
		$\bar{M}_w$	564,800		174,100	83,590	38,400	
		$\bar{M}_w/\bar{M}_n$	1.24		1.30	1.30	1.25	
		σn	223,000		73,630	35,170	15,320	
50	1.0	Fraction number	29	31	34	37	40	
		$\bar{M}_w$	670,000	417,500	206,900	97,380	45,320	
		$\bar{M}_w/\bar{M}_n$	1.23	1.28	1.31	1.32	1.35	
		σn	260,800	172,900	88,190	41,690	19,840	
50	1.5	Fraction number	30		33	36	39	42
		$\bar{M}_w$	614,500		290,800	142,100	65,530	33,520
		$\bar{M}_w/\bar{M}_n$	1.22		1.34	1.35	1.36	1.36
		σn	236,200		126,100	62,480	28,850	14,820
50	2.0	Fraction number	30		33	36	39	42
		$\bar{M}_w$	702,000		307,900	156,000	75,790	34,530
		$\bar{M}_w/\bar{M}_n$	1.31		1.33	1.34	1.42	1.59
		σn	299,100		133,100	68,100	34,550	16,680
80	1.5	Fraction number	30		33	36	39	42
		$\bar{M}_w$	479,500		254,900	122,800	59,810	23,540
		$\bar{M}_w/\bar{M}_n$	1.27		1.28	1.31	1.35	1.42
		σn	195,800		105,300	52,040	26,170	10,740
140	1.5	Fraction number	30		33	36	39	42
		$\bar{M}_w$	450,000		231,700	112,100	51,530	24,350
		$\bar{M}_w/\bar{M}_n$	1.32		1.31	1.37	1.42	1.35
		σn	192,300		98,810	49,760	23,530	10,690

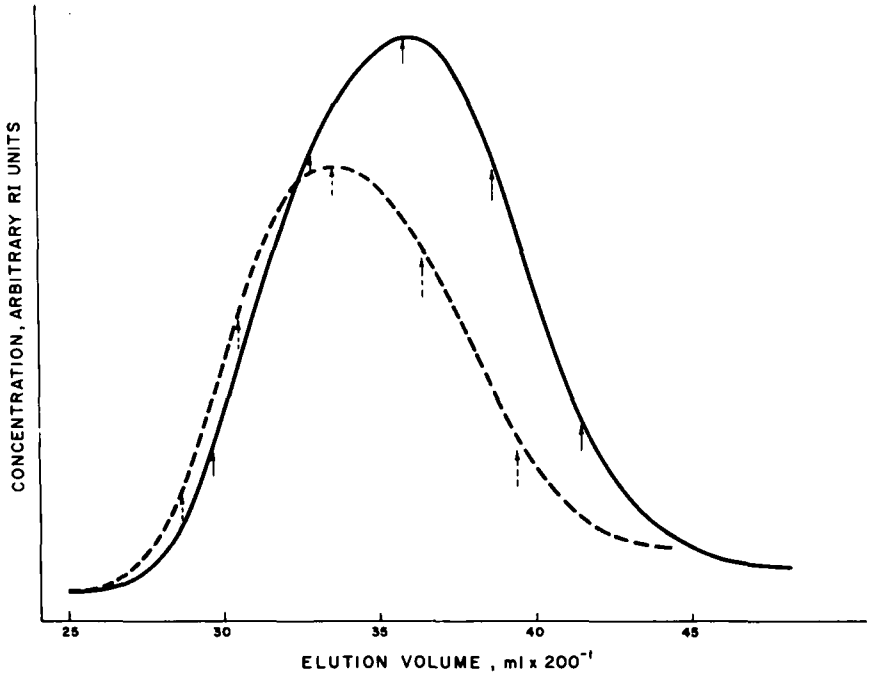

Fig. 3. Chromatograms of Shell crystal-grade polystyrene at two sample concentrations, 0.5 g/100 ml (dotted line) and 2.0 g/100 ml (solid line): solvent, tetrahydrofuran; flow rate, 50 ml/min; injection volume, 100 ml. Arrows indicate where fractions were collected for molecular weight analysis.

There is no change in elution volume with flow rate, for this polymer, over the range of 50 → 140 ml/min. The syphon dump volume was calibrated at each flow rate and was found to be constant in this flow rate range.

Analytical GPC characterizations were carried out using the columns and conditions described in the experimental section. The calibration curve using narrow molecular weight distribution polystyrenes is shown in Figure 5. The chromatograms from the analytical GPC of the preparative GPC fractions were analyzed[13] for $\bar{M}_n$ and $\bar{M}_w$, and the values are shown in Table II. In addition, the standard deviation of the number distribution σN of each fraction is shown.[13] The unfractionated polystyrene had $\bar{M}_w$ = 262,000, $\bar{M}_n$ = 125,300, and $\bar{M}_w/\bar{M}_n$ = 2.09. The data obtained at 50 ml/min flow rate for the four concentrations studied are plotted in Figure 6. The quality of the fractionation, monitored by the experimental $\bar{M}_w/\bar{M}_n$ values, deteriorates as the concentration of 2.0 g/100 ml is reached, when the quality of the lower molecular weight fractions becomes markedly worse. At any given concentration, the higher molecular weight fractions have the smallest polydispersity, with the exception of the lowest concentration studied. The data of Figure 6 are uncorrected for band broadening which increased the measured polydispersities over their true

TABLE III
Values of Resolution Factor h from Analytical GPC Chromatograms

Polystyrene $\bar{M}_w$	True $\bar{M}_w/\bar{M}_n$	Experiment value of $\bar{M}_w/\bar{M}_n$	D	h
670,000	1.06	1.13	0.226	0.40
411,000	1.06	1.10	0.226	0.692
200,000	1.06	1.10	0.226	0.692
97,200	1.06	1.10	0.226	0.692
19,800	1.06	1.15	0.226	0.314

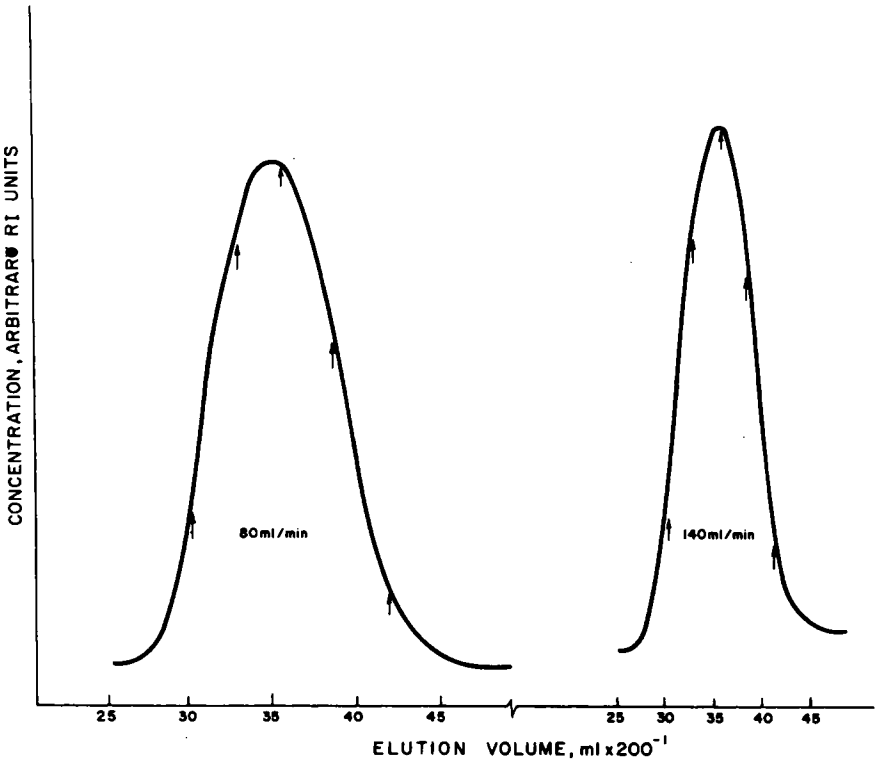

Fig. 4. Chromatogram of Shell crystal-grade polystyrene at two different flow rates, 80 ml/min and 140 ml/min: solvent, tetrahydrofuran; sample concentration, 1.5 g/100 ml; injection volume, 100 ml. Arrows indicate where fractions were collected for molecular weight analysis.

values. Furthermore, this band broadening is molecular weight dependent. In order to obtain the dependence of column efficiency on molecular weight, the effect of band broadening must be considered.

In order to characterize the efficiency of the analytical GPC columns, the chromatograms of the polystyrene standards used to generate the calibration curve were analyzed to generate the differential molecular

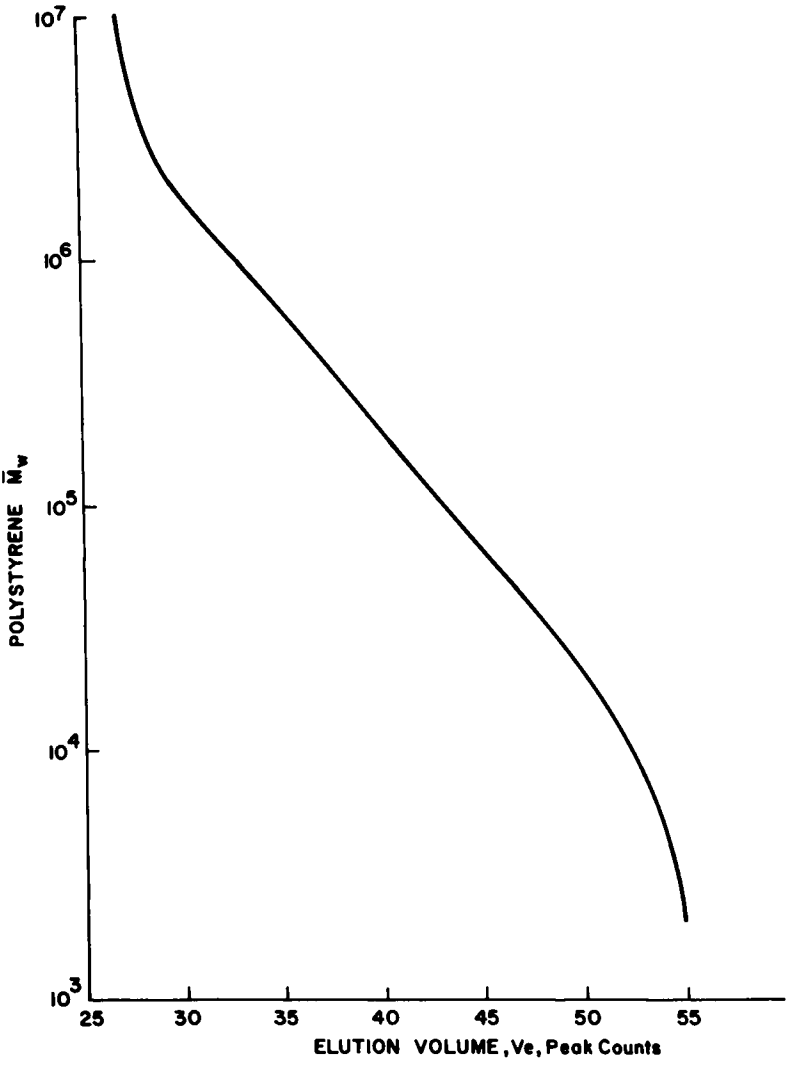

Fig. 5. Calibration curve for analytical GPC columns: polystyrene solutes, tetrahydrofuran solvent; flow rate, 1.5 ml/min; sample concentration, 1 mg/ml; injection volume, 2 ml.

weight distribution and molecular weight averages. Taking the value for $\bar{M}_w/\bar{M}_n$ to be 1.06 the resolution factor[15] h may be calculated from eq. (1)

$$\left(\frac{\bar{M}_w}{\bar{M}_n}\right)_{\text{true}} = \left(\frac{\bar{M}_w}{\bar{M}_n}\right)_{\text{experimental}} \times \exp\left(-D^2/2h^2\right) \qquad (1)$$

where D is the slope of the calibration curve, $\ln M = C - (D \cdot V_e)$.

These values are recorded in Table III, and it is apparent that the columns are highly efficient from a comparison of the true to experimental $\bar{M}_w/\bar{M}_n$ values.

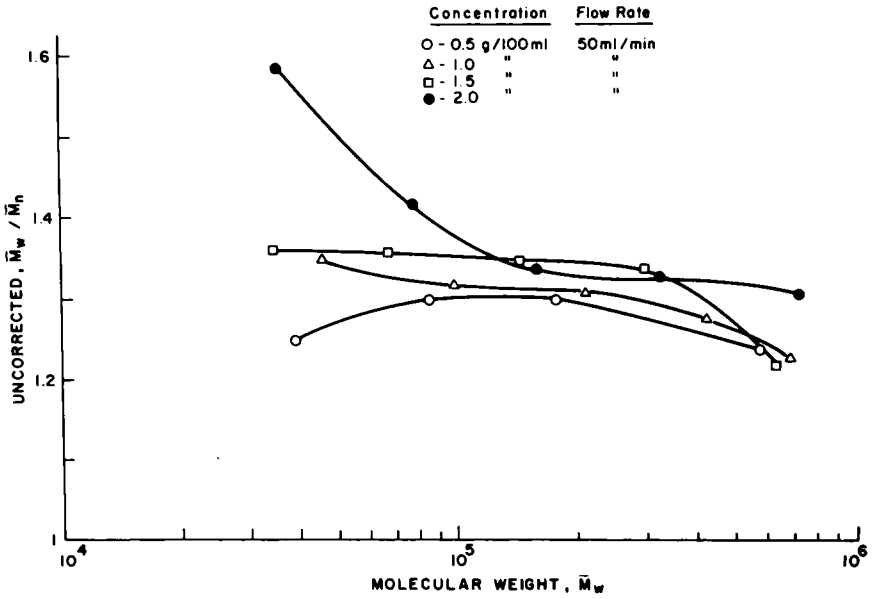

Fig. 6. Uncorrected values of $\bar{M}_w/\bar{M}_n$ from analytical GPC of the preparative GPC fractions plotted vs. molecular weight $\bar{M}_w$.

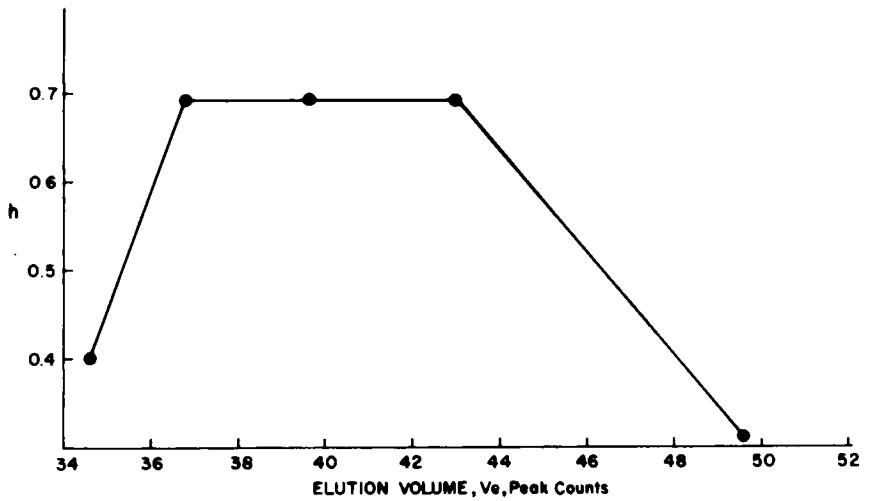

Fig. 7. Resolution factor h plotted vs. elution volume for analytical GPC columns.

The values of h are plotted in Figure 7; it can be seen that h is constant between the elution volumes 36.8 and 43 peak counts. Values outside this range were obtained by connecting the points indicated with straight lines. Reading the appropriate value of h from Figure 7 and using the experimental value of $\bar{M}_w/\bar{M}_n$, eq. (1) may be used to calculate the true value of $\bar{M}_w/\bar{M}_n$ for any sample. These values are shown in Table IV.

TABLE IV

Values of $\bar{M}_w$ and $\bar{M}_w/\bar{M}_n$, Corrected for Band Broadening, for Fractions Obtained from Preparative-Scale GPC Fractionation of Shell Crystal Grade Polystyrene

Flow rate, ml/min	Injected concentration, g/100 ml							
50	0.5	Fraction number	29		34	37	40	
		$\bar{M}_w$	551,000		171,000	81,900	37,400	
		$\bar{M}_w/\bar{M}_n$	1.18		1.26	1.25	1.18	
50	1.0	Fraction number	29	31	34	37	40	
		$\bar{M}_w$	651,200	409,800	203,100	95,500	44,100	
		$\bar{M}_w/\bar{M}_n$	1.16	1.24	1.27	1.27	1.28	
50	1.5	Fraction number	30		33	36	39	42
		$\bar{M}_w$	599,400		285,500	139,500	64,000	32,500
		$\bar{M}_w/\bar{M}_n$	1.16		1.29	1.31	1.30	1.28
50	2.0	Fraction number	30		33	36	39	42
		$\bar{M}_w$	680,900		302,300	153,100	74,200	33,400
		$\bar{M}_w/\bar{M}_n$	1.24		1.28	1.29	1.36	1.49
80	1.5	Fraction number	30		33	36	39	42
		$\bar{M}_w$	470,000		250,200	120,500	58,400	22,600
		$\bar{M}_w/\bar{M}_n$	1.22		1.23	1.26	1.28	1.31
140	1.5	Fraction number	30		33	36	39	42
		$\bar{M}_w$	441,900		227,400	110,000	50,200	23,400
		$\bar{M}_w/\bar{M}_n$	1.27		1.27	1.32	1.35	1.25

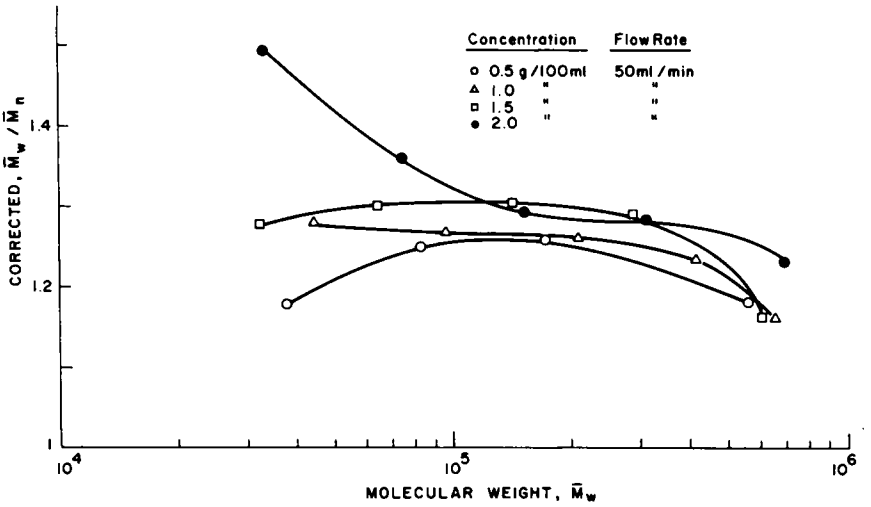

Fig. 8. Values of $\bar{M}_w/\bar{M}_n$, corrected for band broadening, of preparative GPC fractions plotted vs. molecular weight $\bar{M}_w$.

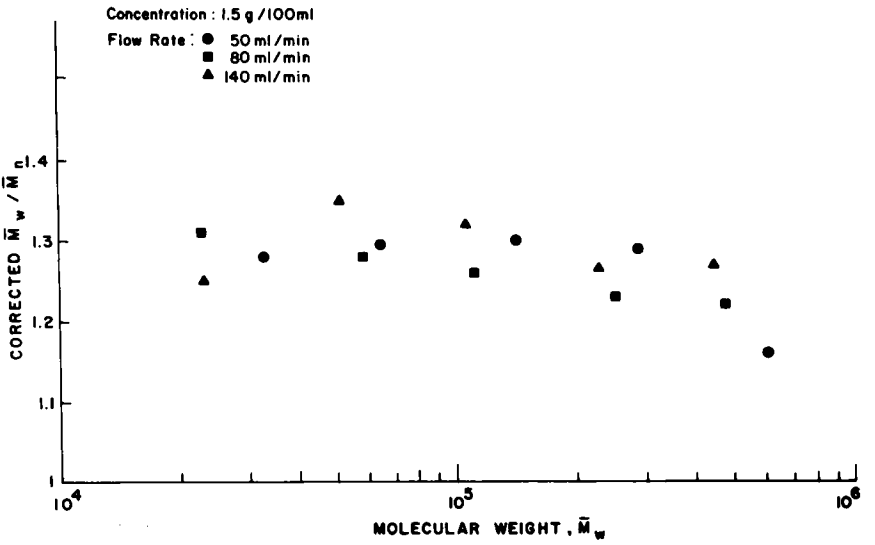

Fig. 9. Effect of flow rate on values of $\bar{M}_w/\bar{M}_n$, corrected for band broadening, of preparative GPC fractions at constant sample load (1.5 g).

In addition, the corrected values obtained for the unfractionated polystyrene are as follows: $\bar{M}_n$ = 127,600; $\bar{M}_w$ = 257,200; $\bar{M}_w/\bar{M}_n$ = 2.015.

The values of $\bar{M}_w/\bar{M}_n$ corrected for band broadening in this manner are plotted in Figure 8 for all concentrations investigated at 50 ml/min flow rate. The trends found in Figure 6 are again seen in Figure 8. The values of the polydispersities of the fractions obtained at a 0.5 g/100 ml concentra-

tion show a maximum in the molecular weight region of $1\text{–}2\times10^5$. This suggests that the polydispersity of the fractions obtained is related to the concentration of that molecular weight in the original sample. It would appear that sharper fractions in the central molecular weight region could be induced by equalizing the concentration of all molecular weights in the original sample. This would be achieved by blending appropriate samples to obtain an approximation to a box distribution. As the sample concentration is increased at constant flow rate, there is a modest increase in the value of $\bar{M}_w/\bar{M}_n$ at intermediate and high molecular weights.

In Figure 9, the values of $\bar{M}_w/\bar{M}_n$, corrected for band broadening, are plotted against $\bar{M}_w$ for three different flow rates: 50, 80, and 140 ml/min. In these experiments, the amount of sample was held constant at 1.5 g. Although there is some scatter in the data, it may be seen that there is very little loss in efficiency with almost a threefold increase in flow rate.

CONCLUSIONS

Polymer concentration has a significant effect on elution volume in a four-column preparative GPC column system. The effect has not been reported before in preparative GPC separations.

The amount of polymer injected into a preparative GPC system has a considerable effect on the polydispersity of the fractions obtained. At the lowest concentration studied, the value of $\bar{M}_w/\bar{M}_n$ was proportional to the amount of polymer in the original sample. This suggests that broad molecular weight distribution polymers may give better fractionations by preparative GPC. At higher concentrations, we have found, in agreement with others, that the polydispersity of the high molecular weight fractions are not affected. The low molecular weight fractions have increasingly higher polydispersities as the injection concentration increases.

The flow rate in preparative GPC is usually set to give the same linear flow velocity as running a 4-ft × $^3/_8$-in.-O.D. (0.035 in. wall) analytical column at 1 ml/min. The results reported here at 80 ml and 140 ml/min shows that the values of $\bar{M}_w/\bar{M}_n$ of the fractions remained constant. Others have arrived at the same conclusion by studying the resolution between two narrow molecular weight distribution polymers. Increasing throughput in the preparative GPC may be achieved by increasing the flow rate up to three times the 50 ml/min flow rate normally used.

No evidence is yet available which would lead to a recommendation of one particular type of packing material. Our results compare with those using a similar-size bank packed with Porasil (Spherosil). No comparable results with a similar-sized bank of Styragel columns are available.

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