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The Use of Porous Glass as a Column Packing for Gel Permeation Chromatography*

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Synopsis

Use of porous glass with a broad pore-size distribution as a column packing for gel permeation chromatography has been investigated. The porous glass is readily available, and columns are packed easily and have excellent mechanical stability. Separations of polystyrene over molecular weight ranges of 500–2,000,000 have been obtained. Porous glass thus appears to be a useful packing material for gel permeation chromatography.

Introduction

Inherent disadvantages of the present organic column packings for gel permeation chromatography (GPC) are solvent selectivity, temperature sensitivity, and mechanical instability. It was recognized by Vaughan[1] that the use of porous glass might avoid these problems. However, his experiments on Corning porous glass No. 7930 (porosity, 40 A.) and crushed, sintered glass (porosity 400,000 A.) showed no separation at all on a benzene solution of a polystyrene with a very broad molecular weight distribution. This failure can probably be explained by the extremely small pore size of one glass and the large pore size of the other. Suitable materials were prepared by Haller[2] by careful heat-treatment and subsequent leaching of an alkali borosilicate glass. The resulting pore sizes were extremely uniform and could be varied between 170 and 1700 A. In another work Haller[3] obtained good separation on mixtures of tobacco mosaic and ring spot virus and on bovine serum. The same packing material was used by Moore and Arrington[4] to fractionate narrow-distribution samples of polystyrene and polyisobutene. They found under ideal conditions that the pore diameter of the glass corresponds to the smallest probable size of the molecules at the upper limit of separation and to the largest probable size of those at the lower limit of the separating range of the column.

Experiments and Results

A series of crushed porous glasses with particle sizes between 80 and 100 mesh have recently become commercially available (Bio-Rad Laboratories,

* Part XIII of a series on column fractionation of polymers.

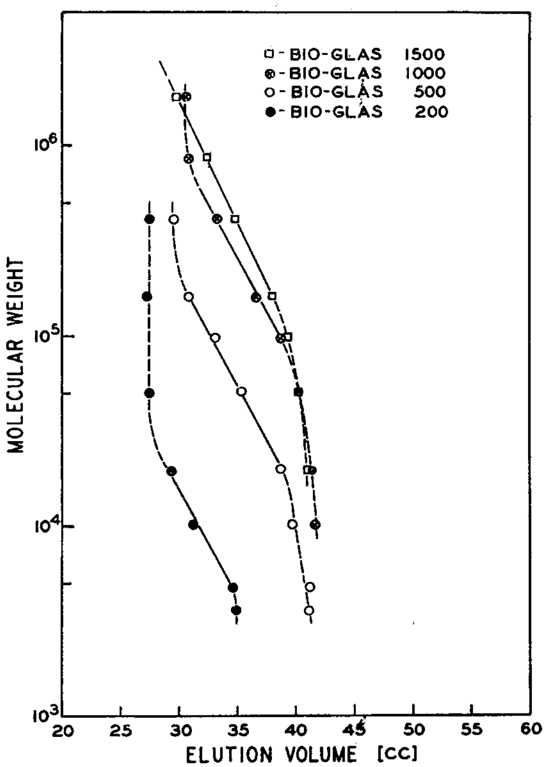

Fig. 1. Calibration for individual glass columns.

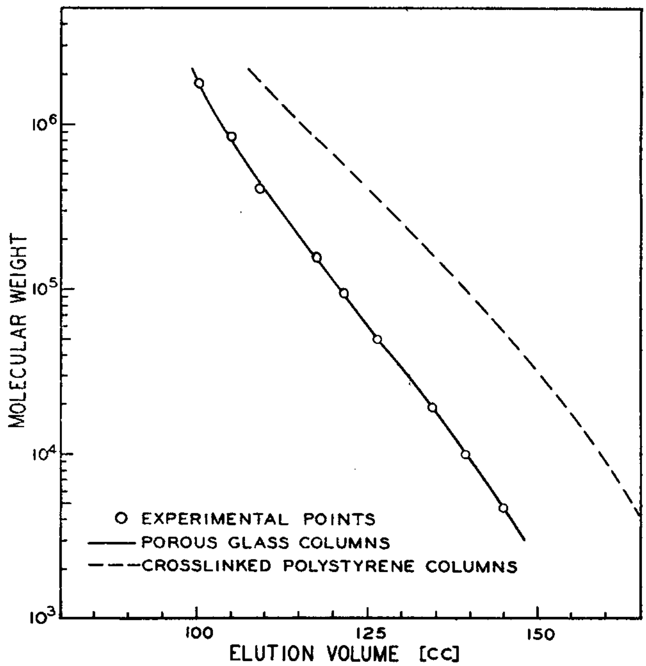

Fig. 2. Porous-glass multiple column.

Richmond, California). Unlike Haller's glasses, they possess a rather broad pore-size distribution. The following samples and lower and upper pore size limits, determined by gas sorption isotherms or mercury porosimetry, given in angstrom units (in parentheses), were used in this study: Bio-Glas 200 (30–280), Bio-Glas 500 (60–300), Bio-Glas 1000 (60–1000), and Bio-Glas 1500 (60–1500). One 4-ft. stainless-steel tube with $^3/_8$ in. outer diameter was dry packed with each of the glasses. The columns were closed off with end plugs, described in the literature,[5] and filled with toluene very slowly under gravity feed. Then they were inserted into a GPC unit operating at 23°C. and purged for several hours with toluene delivered by a special pulseless constant-volume pump.[6] After this a very good baseline was obtained. The pressure drop in the columns was much lower than that in comparable columns packed with polystyrene gel.

Calibrations were carried out with 0.1 wt.-% toluene solutions of narrow-distribution polystyrenes (Pressure Chemicals, Pittsburgh, Pennsylvania). Figure 1 shows the resulting curves for the four individual columns. The linear portions demonstrate polymer separation from molecular weights of 5000 up to 2,000,000. All four columns were then combined in series and again calibrated. The solid line in Figure 2 represents the best fit through the experimental points. It was found that the effect of the arrangement in series can be very closely expressed by an addition of the contributions of the individual columns from Figure 1. The dashed line in Figure 2 represents the calibration curve for a system of four 4-ft. columns filled with crosslinked polystyrene gels with pore sizes of 10^6, 3×10^4, 1.5×10^4, and 200 A. (as indicated by the supplier, Waters Inc., Framingham, Massachusetts). It is seen that the resolution of the two systems is about equivalent.

The performance of the multiple porous-glass column is again shown in Figure 3. The figure represents a trace of an actual chromatogram of a

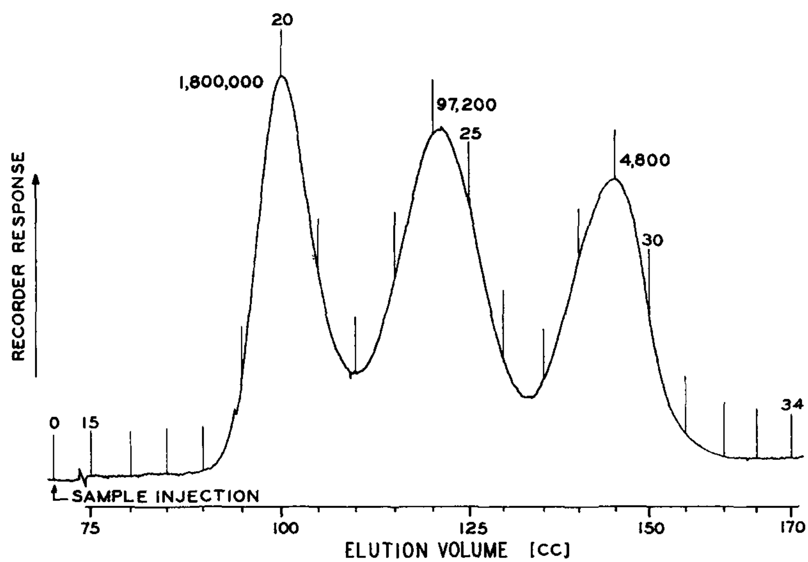

Fig. 3. Chromatogram for four glass columns.

mixture of polystyrenes of molecular weights 1,800,000, 97,200, and 4,800 at a concentration of 0.1% each.

Porous glass is a material with excellent mechanical properties for gel permeation chromatography, and is capable of resolving polymers over a wide molecular-weight range. Columns may be packed easily and require no special precautions to exclude air. Since physical properties such as pore-size distributions may be measured by conventional methods, they may be of interest in studying the mechanism of separation.

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References

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Résumé

On a étudié l'utilisation du verre poreux ayant une distribution assez large, dans les dimensions des pores, comme matériel de remplissage pour colonnes de chromatographie par perméation sur gel. Le verre poreux est facilement disponible, les colonnes sont facilement remplies et possèdent une stabilité mécanique excellente. On a pu séparer des polystyrènes dont les poids moléculaires s'étendaient de 500 à 2.000.000. Le verre poreux semble donc être un matériel de remplissage utile pour la chromatographie par perméation sur gel.

Zusammenfassung

Die Verwendung von porösem Glas mit breiter Porengrössen-Verteilung als Säulenpackung für die Gelpermeationschromatographie wurde untersucht. Das poröse Glas ist leicht erhältlich, die Säulen lassen sich leicht packen und besitzen ausgezeichnete mechanische Stabilität. Auftrennung von Polystyrol im Molekulargewichtsbereich von 500–2.000.000 wurde erhalten. Poröses Glas scheint daher ein brauchbares Packungsmaterial für die Gelpermeationschromatographie zu sein.

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