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APPENDIX A

The determination of pore size distribution

The determination of pore size, pore size distribution, and pore volume are, of course, the three major factors that determine the ability of a spherical bead to be used in a diffusion based separation.

The most common way normally done is to pack the beads in a column and determine what is equivalent to GPC calibration curves.

The GPC columns were the 7.8 by 300 mm stainless steel columns with 0.45 micro frits. The columns were emptied and then washed with acetone, then washed the end frits with acetone and allowed to dry.

The packing material was prepared by making a thin slurry in toluene and stirring to keep in suspension. After 30 to 60 minutes of gentle stirring, allowed the gel to sink and decanted any fines. This process should be continued until all fines have been decanted from the gel.

Then the gel material was made into a thick slurry in toluene and this slurry was pulled by a vacuum to remove all air from around the particle and from within the pore structure of the gel. Allowed the gel to sit, under vacuum, overnight to ensure all air has been removed and the particles were toluene swollen.

First, the column was filled with degassed toluene and then attached as a U-tube-like arrangement. This U tube was filled with degassed toluene and the gel structure and the gel slurry, which would fall to the bottom of the U. This U tube should be connected to the column by means of appropriate fittings that would hold pressure. The outlet of the column to be packed would have a 0.020 inch ID line to a waste container. To the other end of the U tube, by means of fittings, connected your LC pump that had been purged with toluene. Set the pressure cutoff to 5000 psi and set flow to 5 ml/min and pumped the slurry into the column. Continued to allow the LC pump to pump toluene until 50 mls were collected in the waste.

With the column that was packed still full of toluene, filled any void at the top with a known weight of solid glass beads. Determined the weight of beads being used by difference. When the column was full, put the fitting back in place. Put the column in an LC instrument and pumped toluene at a flow of 1.5 ml/min for 2.5 hours - this is equivalent to 10 column volumes.

Reduced the flow to 1.0 ml/min and with the column pumped to a differential refractometer, run a series of styrene narrow dispersity standards.

Injected 100 microliters of 0.07% concentration of narrow dispersity polystyrene standards in toluene from styrene monomer to a 3,000,000 mol wt. material.

Constructed a plot of retention time vs. log mol

wt. of the standard. From the shape of this curve, V_0 and V_t can be determined. Pore column is the difference between V_t and V_0. Using the 4 sigma approach, plate count can be determined on the styrene monomer peak. The pore volume should be reduced to ml/ gram of gel in the column.

This approach should be allowed evaluation of the gel samples that have been synthesized.

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APPENDIX B

The determination of bead density

The liquid displacement method is the most popular method for measuring the true density of the porous beads. The principle of the method is described below.

Equation of pycnometer:

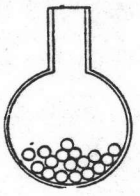
$$P_d = \frac{W_b - W_a}{(W_b - W_a) - (W_c - W_d)} \times L_d \qquad \text{eq. 21}$$

P_d : True bead density

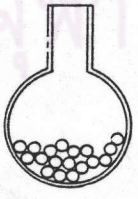
L_d : Liquid dispersion density

W_a, W_b, W_c, W_d are illustrated clearly as follows

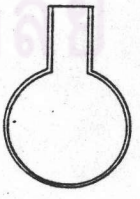
W_a(g) : Weight of the cell



W_b(g) : Total weight of cell and sample



W_c(g) : Total weight of cell, sample and dispersion medium



W_d(g) : Total weight of cell and dispersion medium

Therefore, the true density, P_d, can be calculated from [eq. 21] as above.

VITA

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