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Novel high resolution GPC column Jordi Resolve DVB 5 μm Columns

Introduction

Molecular weight is a critical characteristic of all polymers and strongly influences polymer properties. Most mechanical and rheological properties are a linear function of molecular weight. Molecular weight characterization is therefore essential for the development and manufacturing of polymeric materials.

Gel permeation chromatography (GPC), also known as Size Exclusion Chromatography (SEC), is a well-known technique for determining the molecular weight distribution of macromolecules. In GPC, the separation occurs on the basis of size. Compounds are retained for different periods of time based upon their access to the porous structure of the chromatographic packing. The smaller analyte molecules enter the pores more easily and therefore spend more time in these pores, resulting in increased retention time. Conversely, larger analyte molecules spend less time in the pores and are eluted quickly. Thus, chromatographic separation is based on hydrodynamic volume (size in solution).

During standardized GPC, sample components are dissolved in a suitable solvent, chromatographically separated based on molecular size, detected using a Refractive Index detector and compared to standards of known molecular weight. Absolute molecular weight determinations can also be performed by coupling light scattering and viscometry detection to a GPC separation.

In GPC the mobile phase serves to dissolve the sample and carry it through the column. Under ideal conditions, there is no interaction between the analyte molecules and the stationary phase. In order to obtain a purely size based separation, a suitable combination of column stationary phase and mobile phase is necessary. Jordi Labs has recently developed a novel packing material for GPC systems. This column packing is based on 100% divinylbenzene providing enhanced mechanical stability and utilizes a new synthetic process which results in a monodisperse polymer column packing. It is well known that broad particle size distributions in particle based columns produces variations in packing density, lowers column resolution, reduces the column permeability and generates high back pressure. New generation Jordi GPC columns prepared with monodisperse 100% divinylbenzene particles with precisely controlled particle diameter and finely controlled pore structure provide high efficiency, high separation capacity and low back pressure with greater bed stability. Scanning electron micrographs and

particle size distribution of 5 μ m macroporous column packing material with 10³ A pore sizes are shown in **Figure 1**. The uniform size distribution and perfect spherical shapes are clearly seen.

To maximize GPC resolution, the key is to use a column containing the maximum number of pores of the desired size to separate the molecular weight range of interest. New generation individual pore size Jordi Resolve columns 5 µm (7.8 mm ID x 300 mm L) provide high resolution in specific molecular weight ranges as shown in Figure 2. The linear part of each calibration curve defines the molecular weight range of each individual porosity column. To cover a wider range of molecular weight with a constant resolution for the analysis of polydisperse or unknown materials, Jordi Labs designed mixed bed 5 µm Resolve columns (7.8 mm ID x 300 mm L). Mixed bed Resolve column allows separation over the molecular weight range from 160 to 3,000,000 g/mol (PS equivalent) with a coefficient of determination (R²) of 0.999, as shown in Figure 3, and an industry leading specification of 70,000 theoretical plates. Lower slope of the calibration curve (13%) of Resolve mixed bed column compared to the competitor column of the same particle size and length shows that Resolve column provides better separation with more accurate molecular weights (Figure 3). Clearly, the high pore volume of Jordi Resolve columns provides increased resolution as indicated by the chromatograms in Figure 4. Accurate blending of a large number of individual pore size gels results in a wide linear molecular weight range and smooth peak shapes. Analyses of broad distribution polymers obtained using Jordi Resolve column, as illustrated in Figure 5, show high resolution without any dislocations or shoulders.



Figure 1. Scanning electron micrograph and particle size distribution of Jordi Resolve 5 μ m column packing material with 10³ Å pore size.



Figure 2. Calibration curves of individual pore size Jordi Resolve 5 μm columns. Mobile phase: tetrahydrofuran, flow rate: 1.0 mL/min, sample: polystyrene standards, injection volume: 100 μL



Figure 3. Calibration curves of three column set of Jordi Resolve Mixed Bed 5 μ m column (7.8 mm ID x 300 mm L) and the competitor column. Mobile phase: tetrahydrofuran, flow rate: 1.0 mL/min, sample: polystyrene standards, injection volume: 100 μ L.



Figure 4. Overlay of chromatograms of 5 polystyrene standards obtained with three column set of Jordi Resolve (7.8 mm ID x 300 mm L) and the competitor column. Mobile phase: tetrahydrafuran , flow rate: 1.0 mL/min, injection volume: 100μ L.



Figure 5. Analyses of broad distribution polymers. Columns: 3x Jordi Resolve mixed bed 5 μ m (7.8 mm ID x 300 mm L), mobile phase: tetrahydrofuran, flow rate: 1.0 mL/min, injection volume: 100 μ L.