High Performance GPC Analysis for Reliable Polymer Characterization

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Introduction

Both synthetic and natural polymers have application across a wide range of end uses including engineering polymers for manufactured goods, food additives, pharmaceutical excipients and drug-delivery agents. The properties and processability of a polymer depend greatly on its molecular weight distribution and monitoring and control of this parameter is key to product performance. Gel permeation chromatography (GPC) is a well-established technique for the characterization of polymer molecular weight distribution (MWD). The accuracy and precision of the MWD determined from a GPC experiment rely on the performance of the whole system.

Figure 1: Chromatogram of polystyrene standards (Mp 7 500 000, 841 700, 148 000, 28 500, 2930).

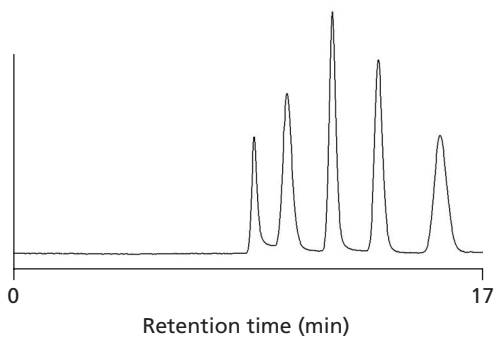
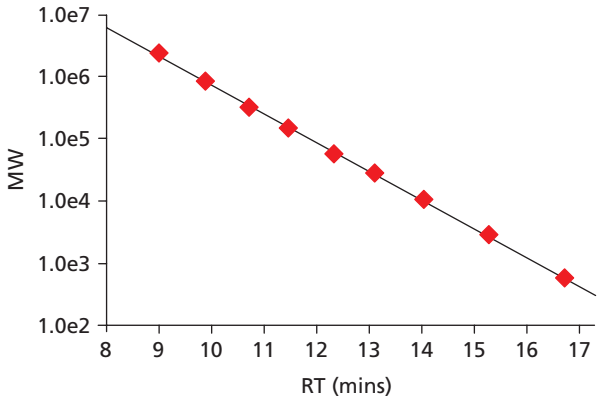


Figure 2: Overlay of five consecutive narrow standards calibration plots.



Experimental

The PL-GPC 20 is an integrated system specifically designed for routine GPC measurements at ambient temperature, which as standard comprises a precision solvent delivery system, choice of manual or automatic injection, and high-performance differential refractive index (RI) detector. Operation of the system is performed via an intuitive Windows-based control software package and data from the RI detector is acquired and analysed using Cirrus GPC software. A wide variety of both organic (PLgel) and aqueous (PL aquagel-OH) GPC columns can be selected according to application. The conditions employed for this set of experiments were: columns = 2 × PLgel 5 µm MIXED-C, 300 × 7.5 mm; eluent = THF; flow-rate = 1.0 mL/min.

Results

The RI detector displays both high sensitivity and excellent baseline stability for a wide variety of organic GPC solvents and aqueous-based eluents. This is illustrated in Figure 1 which shows a

Figure 3: Results and MWD overlay for five injections of a commercial polystyrene.

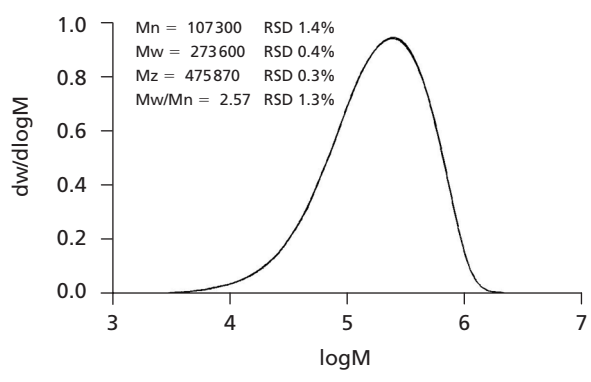
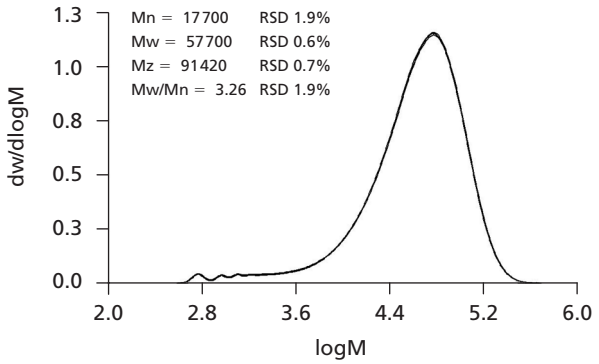


Figure 4: Results and MWD overlay for five injections of a commercial polycarbonate.



separation of five narrow polydispersity polystyrene standards (concentration 0.1%, injection volume 100 µL), a typical chromatogram generated as part of the column calibration procedure in GPC.

The flow-rate precision of the solvent delivery system is key to achieving the most reliable GPC determinations. The PL-GPC 20 delivers flow reproducibility of better than 0.10%, resulting in repeatable calibration curves and accurate polymer molecular weight data. Figure 2 shows an overlay of five consecutive GPC calibrations generated using a series of nine narrow polydispersity polystyrene standards. The retention times, uncorrected for flow-rate variation, of all of the standards are identical from one calibration to the next giving a perfect overlay of the five graphs indicating excellent repeatability.

As a further illustration of the repeatability of the system, Figures 3 and 4 show overlays of five molecular weight distribution plots calculated from five consecutive injections of two different commercial polymers, a polystyrene and a polycarbonate. The molecular weight averages calculated for each injection and the mean values and % variation are indicated alongside each plot. In both cases the repeatability of the determined values is better than 2%, which is well within the generally accepted limit for GPC of 3–5%.

Conclusions

The PL-GPC 20 is an integrated GPC system suitable for polymer characterization with ambient temperature operation. The system permits polymer MWD to be measured with a high level of accuracy and precision. The optional autosampler would be a valuable addition to the system for those laboratories handling large numbers of samples.

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