

# Instrument Setup for Fast GPC/SEC

## Technical Overview

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### Abstract

Proper column choice and instrument setup together can vastly improve the speed and data quality that can be achieved when performing gel permeation chromatography (GPC)/size exclusion chromatography (SEC). Agilent manufactures a full range of consumable parts that enable the best possible results achieved with various types of instruments.

### Introduction

Gel permeation chromatography (GPC) is an important technique for determining the molecular weight (MW) distribution of a polymer, and for comparing batch-to-batch polymer quality. With laboratories under increasing pressure to increase efficiency and reduce costs, Agilent is committed to offering GPC users innovative solutions to increase the speed and quality of their analyses.

Optimal system setup is key to reduce the cost per analysis for labs performing GPC, but systems vary widely in performance. This technical overview focuses on the different strategies to maximize performance on low and high dispersion instruments.

Low dispersion instruments, in combination with high efficiency columns, offer the highest possible speed and resolution. The columns separate samples quickly while the system transports the analytes from injector to column to detector without allowing these narrow peaks to broaden.

In practice, many analysts must use instruments that have too high dispersion to benefit from high efficiency columns. To help these customers improve their analysis, Agilent has designed PL Rapide columns to counter the dispersion in their instrument. Agilent PL Rapide columns offer a drop-in solution to significantly improve performance on systems that were previously dispersion-limited.



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## Low Dispersion Systems

Modern HPLC systems, such as the Agilent 1260 Infinity II GPC/SEC system, have been optimized to perform high speed, high-resolution chromatography by reducing dispersion in all internal components of the system.

### System

Certain key components are necessary for fast GPC, including:

- **Detector**  
Small flowcell detectors are critical to prevent dispersion. The Agilent 1290 Infinity II Refractive Index Detector (p/n G7162B) is equipped with an ultralow dispersion microflow cell, making it ideally suited for most fast GPC applications.
- **Capillary**  
Significant diffusion can occur in the capillary. This diffusion can be minimized with the use of short, narrow capillaries. A capillary with a 75  $\mu\text{m}$  internal diameter is recommended, such as an Agilent stainless steel capillary (p/n 5067-4783).
- **Autosampler**  
Small injection volumes and low dispersion plumbing along the entire sample path are essential for a fast GPC analysis. All the necessary components can be found in the ultralow dispersion kit for Agilent 1290 Infinity LC series (p/n 5067-5189).
- **Fittings**  
It is critical that capillaries are swaged properly to prevent forming voids or leaks. The Agilent award-winning A-Line fittings perfectly seat the capillary in the column at least 200 times without reswaging [1].



Figure 1. The optimized system for GPC. An Agilent 1260 Infinity II GPC/SEC system.

## Columns

Once the liquid chromatography system itself is optimized, high efficiency columns are essential for achieving high-speed GPC.

Agilent PlusPore columns have been optimized to offer the highest efficiency over the most common molecular weight ranges. PlusPore columns are packed with particles that have a very high pore volume to provide significantly increased resolution, compared to a conventional GPC column set.

Table 1. Agilent PlusPore columns.

Column	Molecular weight range (g/mol)	Typical efficiency (p/m)	Max temperature	Dimensions	p/n
PolyPore	200 to 2,000,000	>60,000	150 °C	7.5 × 300 mm	PL1113-6500
				4.6 × 250 mm	PL1513-5500
				2.1 × 250 mm	PL1913-5500
ResiPore	Up to 500,000	>80,000	110 °C	7.5 × 300 mm	PL1113-6300
				4.6 × 250 mm	PL1513-5300
				2.1 × 250 mm	PL1913-5300
MesoPore	Up to 25,000	>80,000	110 °C	7.5 × 300 mm	PL1113-6325
				4.6 × 250 mm	PL1513-5325
				2.1 × 250 mm	PL1913-5325
OligoPore	Up to 3,300	>55,000	110 °C	7.5 × 300 mm	PL1113-6520
				4.6 × 250 mm	PL1513-5520
				2.1 × 250 mm	PL1913-5520

## Results

### High-resolution analysis

Oligopin is a commercial extract from pine bark that contains a complex mixture of high MW condensed polyphenols. Previous analysis with a traditional single pore column stacks provided limited MW and composition information.

Replacing the four individual pore size columns with a stack of three Agilent OligoPore columns reduced runtime, and gave a drastic increase in resolution (Figure 2). This increase in resolution revealed the individual compounds and their MW values, which could now be compared between batches.

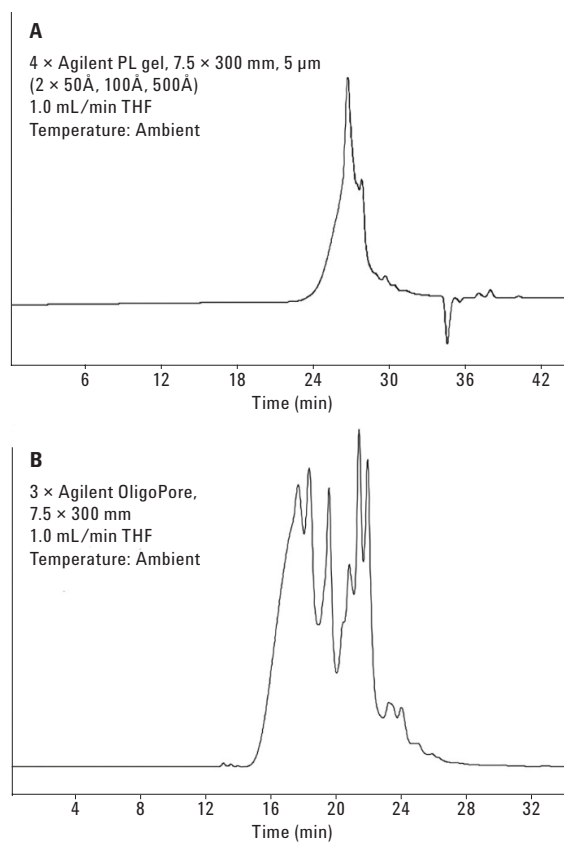


Figure 2. Increased speed and drastic resolution improvement of an Oligopin (commercial polyphenol extract) sample on the low dispersion Agilent 1260 Infinity GPC/SEC system using Agilent OligoPore columns.

### High-speed quality control

In the quality control of simple condensation polymers such as epoxy resin, the content of oligomers, additives, and residues is the primary concern.

The Agilent MesoPore column is ideally suited for high-throughput analysis of these resins, since its 3  $\mu\text{m}$  particle diameter operates at high flowrates with minimal resolution loss.

Figure 3 shows the separation of a commercial epoxy resin using two MesoPore columns, which generated all necessary data for quality control analysis, with a runtime of under 5 minutes.

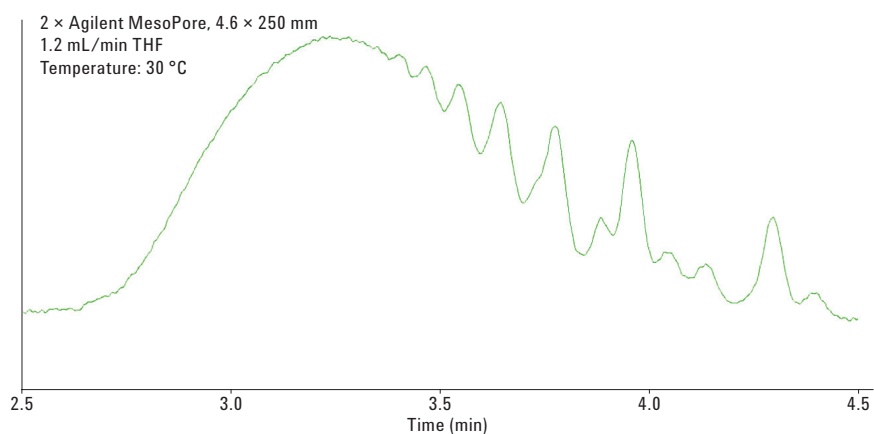


Figure 3. Fast epoxy analysis (Epikote 1004; commercial epoxy resin;  $M_n = 1480$  + oligomers) on the low dispersion Agilent 1260 Infinity II GPC/SEC system.

## High Dispersion Systems

Many components are impossible to miniaturize for reduced dispersion, including light scattering detectors, viscometers, and high-temperature hardware.

Significant dispersion can also result from the use of older components such as autosamplers and large flowcell detectors, as well as capillaries and fittings.

In these cases, the normal performance improvement from using high efficiency columns is lost, since peaks are broadened by the system.

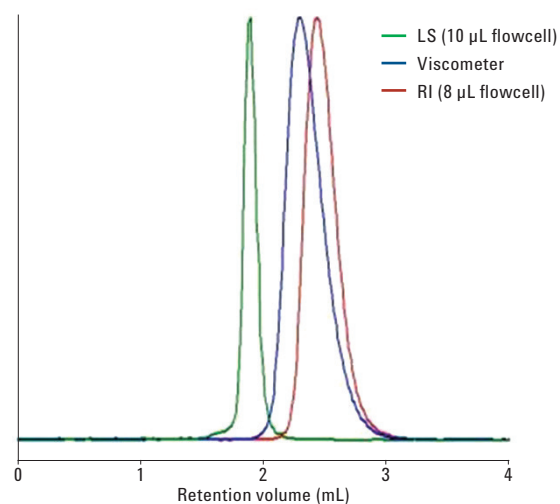


Figure 4. Progressive peak broadening as a commercial polycarbonate resin sample flows through three common GPC detectors: a light scattering detector (green), a viscometer (blue), and a refractive index detector (red).

## Agilent PL Rapide columns – a solution for high dispersion

Agilent PL Rapide columns offer high speed and resolution on high dispersion systems by combining high efficiency PLgel media with wide column diameters that allow high flowrates (1.5–3.0 mL/min).

The high efficiency media generates narrow peaks, while the elevated flow rate minimizes peak broadening as the analyte travels through the high dispersion components of the system.

The result is a significant increase in speed and resolution even on a high dispersion system. Interestingly, the reduced analysis time compensates for the higher flowrate, so that solvent savings of over 50% per analysis are possible.

Table 2. Agilent PL Rapide columns.

Description	Molecular weight range (g/mol)	Guaranteed efficiency (plates/m)	Max temperature	Dimensions	p/n
PL Rapide H	500 to 10,000,000	>35,000	220 °C	100 × 10 mm	PL1113-3100
				150 × 7.5 mm	PL1013-2100
PL Rapide M	200 to 2,000,000	>60,000	150 °C	100 × 10 mm	PL1013-2500
				150 × 7.5 mm	PL1113-3500
PL Rapide L	200 to 500,000	>80,000	110 °C	100 × 10 mm	PL1013-2120
				150 × 7.5 mm	PL1113-3300
PL Rapide F	Up to 3,300	>55,000	110 °C	100 × 10 mm	PL1013-2300
				150 × 7.5 mm	PL1113-3120

## Results

### Maximizing throughput on an older system

Kratons are styrene-based polymers used for rubbers and elastics. A customer method for this analysis required six individual pore size columns, and took over 70 minutes to achieve their resolution requirements.

Direct replacement of the customer's columns with higher pore volume Agilent PLgel columns brought the analysis time down to 45 minutes, as shown in Figure 5. However, the runtime and peak shape were still suboptimal.

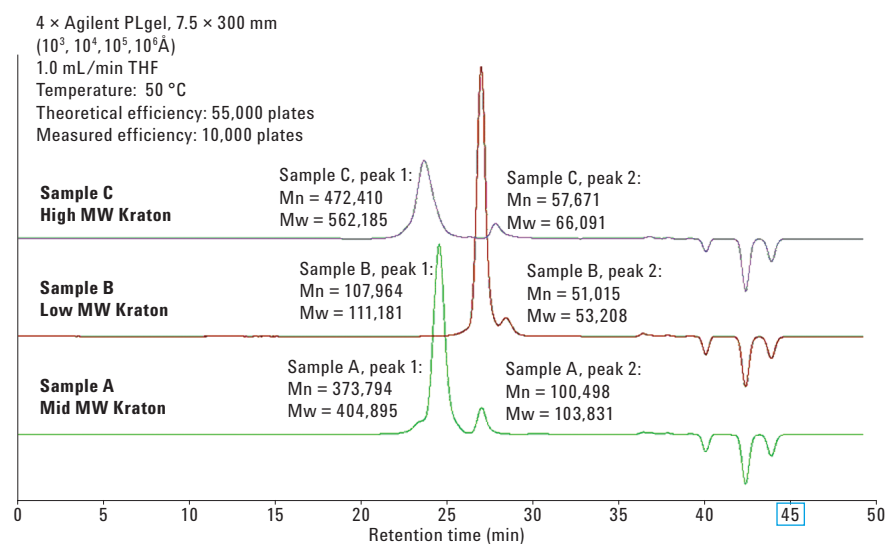


Figure 5. The traditional single pore column stack gives poor efficiency, noticeable shoulders, and a long runtime. Sample: Kratons (various); commercial polystyrene copolymer.



As shown in Figure 6, a 30% improvement in efficiency and 70% drop in runtime is achieved by using Agilent ResiPore columns. The wide MW range prevents dislocations and shoulders, while the 3  $\mu\text{m}$  particles offer much higher efficiency over 5  $\mu\text{m}$  particles.

Despite these improvements, more than 60% of the theoretical efficiency has been lost to instrument dispersion.

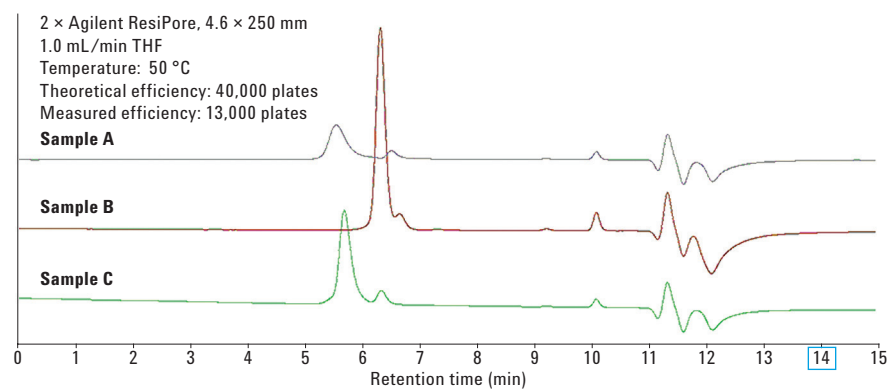


Figure 6. High efficiency Agilent ResiPore columns improve resolution, and significantly reduce runtime, but most efficiency is lost due to the high dispersion instrument.

Figure 7 shows the improvements in the analysis using Agilent PL Rapide L columns. The 1.5 mL/min flowrate boosts efficiency even further, while consuming far less solvent than the original analysis.

By changing to the PL Rapide L columns, the analysis ran five times faster, and the efficiency was more than doubled, without making any changes to the instrument.

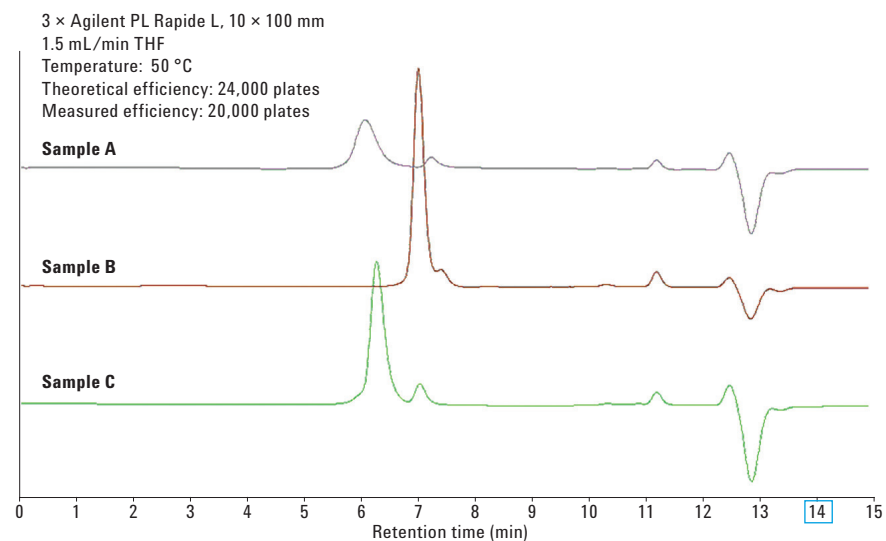


Figure 7. Agilent PL Rapide columns minimize losses to instrument dispersion, and achieve the highest efficiency. Despite higher flowrates, PL Rapide columns consume 50% less solvent than the single pore stack.

## Conclusion

Agilent strives to provide its customers with cost-effective improvements to their day-to-day laboratory operations. Time savings, in particular, are key to maximizing productivity.

In this example, Agilent offers the leading solution in GPC/SEC analysis with its Agilent 1260 Infinity II GPC/SEC system and Agilent PlusPore columns. Meanwhile, for customers who are limited to a higher dispersion system, the Agilent PL Rapide line is a drop-in solution that offers tremendous improvements over older technologies.

By combining expertise, innovation, and a comprehensive family of GPC/SEC columns, calibrants, and instruments, Agilent is able to offer the optimal solution to its customers, regardless of sample, instrument, or experience level.

## Reference

1. *Agilent A-Line UHPLC Fittings*; Application note, Agilent Technologies, Inc. Publication number 5991-5525EN, **2015**.

## For More Information

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