

Low molecular weight resins - Analysis of low molecular weight resins and prepolymers by GPC/SEC

Application compendium

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The Measure of Confidence



Agilent Technologies

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Polymer Laboratories was formed in 1976 to offer high quality columns, standards, instruments, and software for GPC/SEC. For over 30 years the company developed many market-leading products, including PLgel, PL aquagel-OH, PlusPore, PLgel Olexis, PolarGel columns, and EasiVial standards. Built on advanced in-house manufacturing technology, PL's products have the highest reputation for quality and performance, backed up by world-class technical and applications support.

With the acquisition of PL, Agilent offers an even wider range of GPC/SEC solutions for all types of polymer characterization of synthetic and bio-molecular polymers, with options for conventional GPC all the way up to complex determinations using multi-column and multi-detection methods.

Low molecular weight resins

Introduction

The term resin is used to describe materials manufactured by the addition polymerization of reactive monomers, often accompanied by the elimination of a small molecule. These synthetic methodologies result in materials with a relatively low molecular weight and a wide polydispersity, often containing oligomers and a considerable monomer content. A feature of these polymers is the presence of reactive groups at the end of the polymer and oligomer chains, and many such materials are used as prepolymers that can be further reacted to form new products.

Gel permeation chromatography/size exclusion chromatography (GPC/SEC) is a well-known technique for assessing the molecular weight distribution of polymers, a parameter that influences many physical properties. For example, the molecular weight of resin materials determines their physical state and their accessibility to further reactions. Characterizing and understanding the molecular weight distribution of resin materials is therefore key to their performance.

However, the polymers produced by condensation polymerizations tend to be highly functional and contain reactive groups, and many classes of material grouped by their reactive functionality, for example polyurethanes, have widely differing chemical structures. As a result, analysis of these materials by GPC/SEC can be challenging due to the possibility of interactions occurring between the packing material contained in the GPC/SEC column and structural elements of the material.

This application compendium discusses the analysis of low molecular weight resins and highlights those columns best suited for their analysis. A series of example analyses illustrate the quality of data that may be obtained.

Agilent produces GPC/SEC columns, standards and instruments that are ideally suited to the analysis of resins and low molecular weight prepolymers.

Agilent's GPC/SEC columns are ideal for applications that rely on extremely reproducible analysis, such as quality control environments in resin manufacturing. Agilent's columns include ranges suitable for use in organic and aqueous eluents, solvent mixtures and high polarity organic eluents, covering the requirements of the diversity of resins.

The column range includes products tailored to the analysis of low molecular weight materials, and includes high resolution columns, such as the OligoPore and PLgel 3 μm 100Å columns, that are designed for the analysis of oligomeric samples. With extensive options in particle size and pore size, Agilent's columns can be selected to match the molecular weight of the material under investigation simply, thereby ensuring that the best quality of data is obtained from the GPC/SEC experiment.

Agilent's range of integrated GPC/SEC instrumentation covers a temperature range from ambient to 220 °C. Agilent also produces a range of polymer standards to complement resin analysis.

These instruments allow all forms of the GPC/SEC experiment to be performed and can be used to analyze the complete range of resin materials, including those that require analysis in unusual solvents. Multiple detection options can be included in the instruments, such as light scattering and viscometry, and dedicated analysis software is available that allows the properties of resins to be analyzed in detail.

Characterization of low polarity resins

Low polarity resins may be analyzed on polystyrene/divinylbenzene columns in typical GPC solvents, such as THF.

The PlusPore series of columns has been specifically designed for *high resolution* GPC. These packing materials are based on the industry-standard, highly crosslinked polystyrene/divinylbenzene (PS/DVB) packing material, for the widest applicability and solvent compatibility. Each is made using a novel polymerization process to produce particles which exhibit a specific, controlled pore structure for optimum GPC performance.

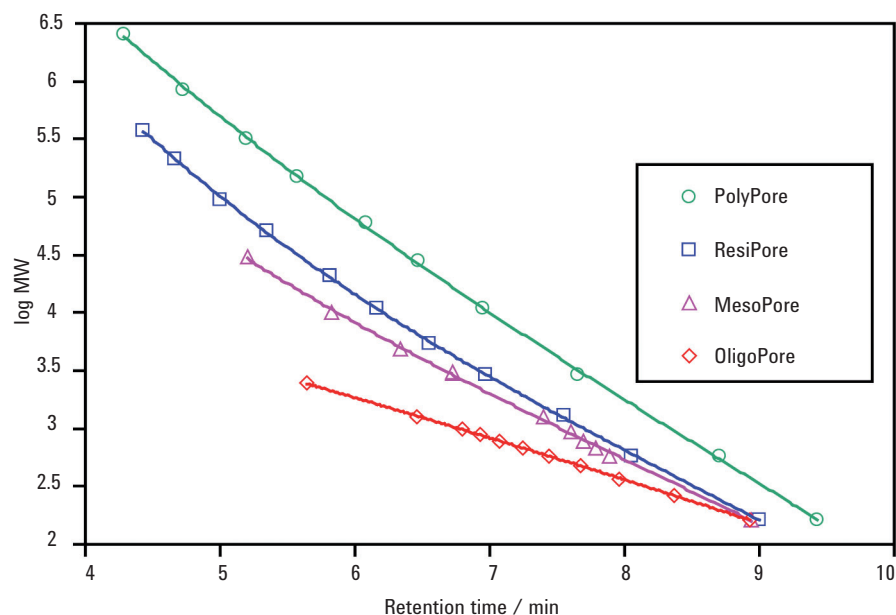


Figure 1. PlusPore calibration curves showing the resolving ranges and near linear calibrations of the columns

The ideal choice for polymer analysis

For high resolution polymer analysis, the PolyPore, ResiPore, MesoPore and OligoPore columns of the PlusPore product range exhibit a wide pore size distribution with near linear calibration curves covering an extended molecular weight range. These so-called 'multipore' structures have increased pore volume compared to regular PS/DVB packing materials. This results in very high resolution GPC columns designed for specific application areas. The highly crosslinked porous particles provide excellent chemical and physical stability and permit easy transfer across the full range of organic solvents with little change in the shape of the calibration curve or the efficiency of the columns. As this 'multipore' column technology does not require the combination of individual pore size packing materials, the result is high accuracy and precision *without* any artefacts in the shape of the molecular weight distribution (MWD).

Features and benefits of the PlusPore range

- High pore volume, high resolution - improved separations and efficiency
- Wide pore size distribution, optimized separation range - separates broad range, multiple usage
- Full solvent compatibility - easy transfer across range
- No MWD dislocations - maximized productivity

The composition of oligomers in resins is of great commercial importance, as is the determination of residual monomer in the quality control of polymers. These low molecular weight samples are routinely characterized by gel permeation chromatography (GPC). Ideally, separation of discrete components is required in order to identify and quantify specific components of interest. In order to achieve this, small particle size packings are used to produce high resolution separations. The MesoPore column, with an exclusion limit of 25,000 molecular weight, has a guaranteed minimum efficiency of 80,000 plates/meter. Typical application chromatograms are shown in Figures 2 to 6.

Sample: Isocyanate
Columns: 2 x MesoPore,
300 x 7.5 mm
(Part No. PL1113-6325)
Eluent: THF (stabilized)
Flow Rate: 1.0 mL/min
Detector: RI

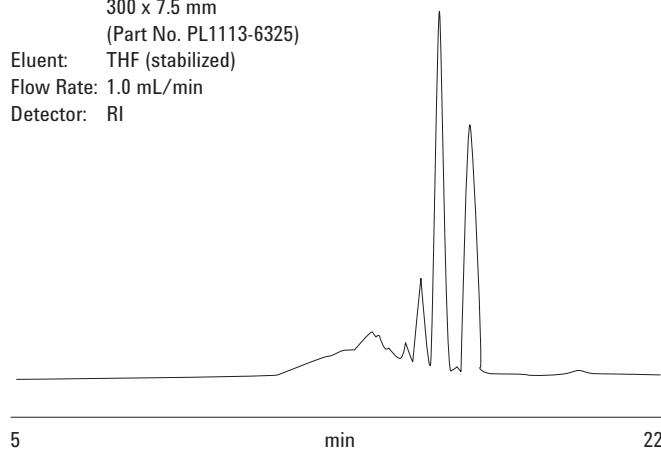


Figure 2. Chromatogram of an isocyanate sample showing polymer and oligomeric detail

Sample: Polyol
Columns: 2 x MesoPore,
300 x 7.5 mm
(Part No. PL1113-6325)
Eluent: THF (stabilized)
Flow Rate: 1.0 mL/min
Detector: UV, 240 nm

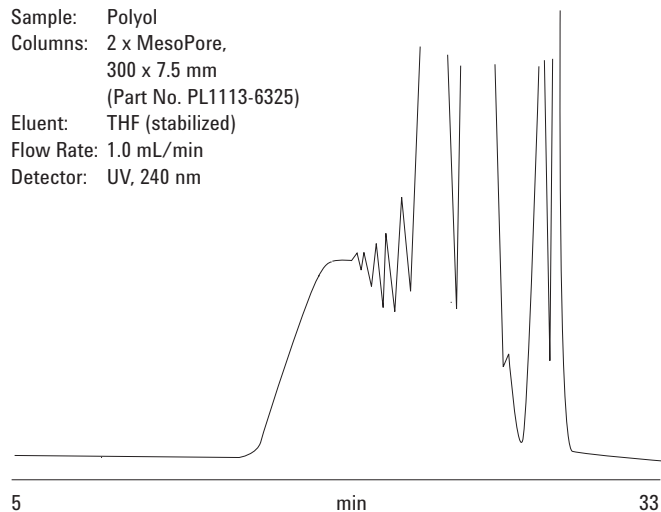


Figure 3. Chromatogram of a polyol sample showing the presence of large amounts of oligomers

Columns: 2 x MesoPore, 300 x 7.5 mm
(Part No. PL1113-6325)
Eluent: THF
Flow Rate: 1.0 mL/min
Inj Vol: 20 µl
Detector: RI

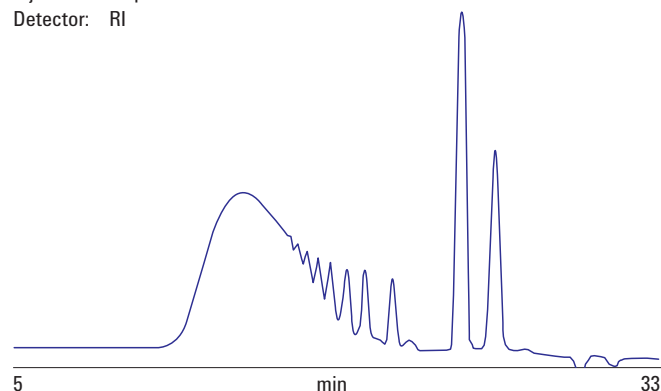


Figure 4. Chromatogram of a polyurethane sample with oligomers and residual monomers

Columns: 2 x MesoPore, 300 x 7.5 mm
(Part No. PL1113-6325)
Eluent: THF
Flow Rate: 1.0 mL/min
Inj Vol: 20 µl
Detector: RI

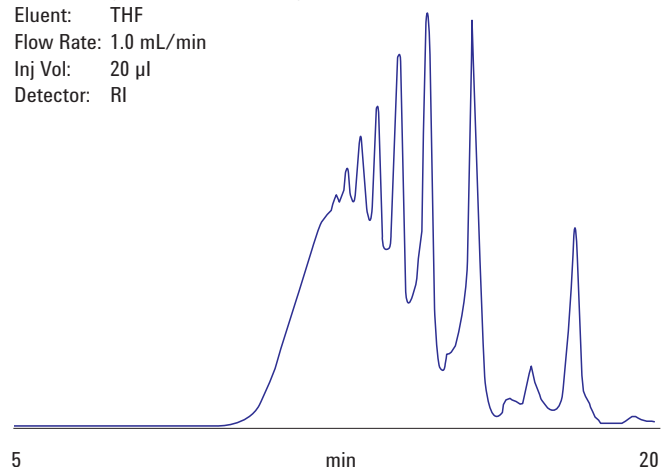


Figure 5. Chromatogram of an epoxy resin sample showing oligomers dominating the distribution

Columns: 2 x MesoPore, 300 x 7.5 mm
(Part No. PL1113-6325)

Eluent: THF

Flow Rate: 1.0 mL/min

Inj Vol: 20 µl

Detector: RI

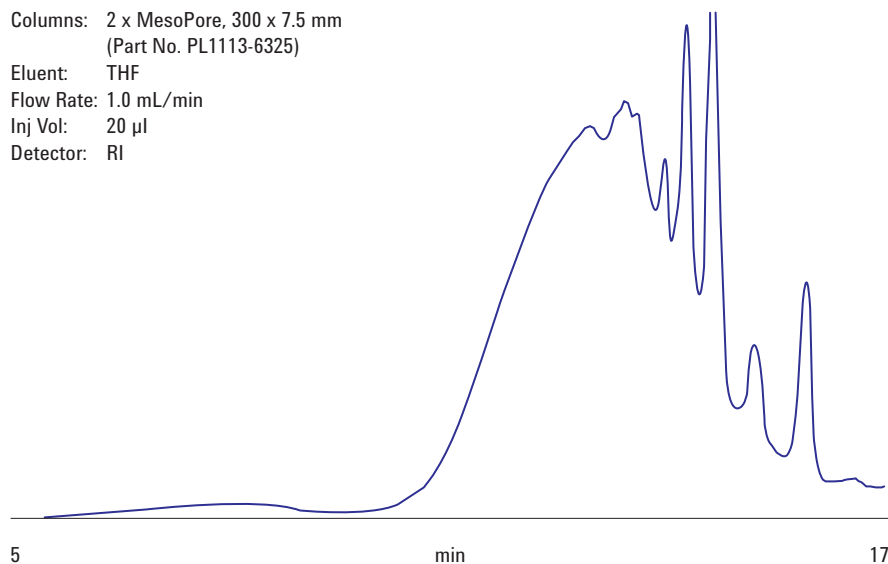


Figure 6. Chromatogram of a polyesterimide sample with a complex distribution of polymer and oligomer content

For higher molecular weight resins, the determination of molecular weight distribution is a primary objective in GPC analysis and columns with a broader resolving range are required. The ResiPore column has been specifically designed for such applications where material above 400,000 molecular weight is unlikely to be present. Figures 7 to 10 illustrate typical application chromatogram.

Sample: Paint resin

Columns: 2 x ResiPore, 300 x 7.5 mm
(Part No. PL1113-6300)

Eluent: THF (stabilized)

Flow Rate: 1.0 mL/min

Detector: RI

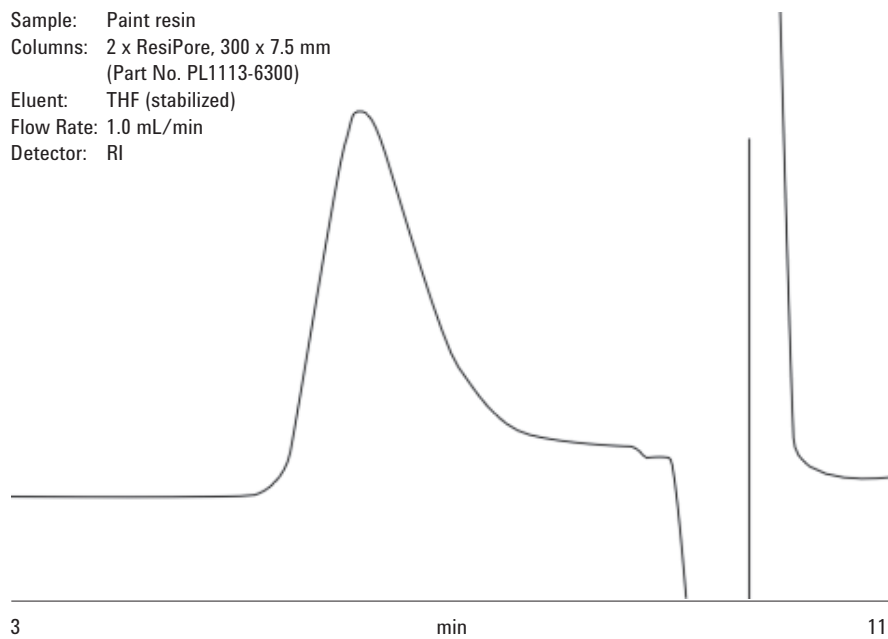


Figure 7. Chromatogram of a paint resin sample showing low molecular weight content with the polymer

Columns: 2 x ResiPore, 300 x 7.5 mm
(Part No. PL1113-6300)

Eluent: THF

Flow Rate: 1.0 mL/min

Inj Vol: 20 µl

Detector: RI

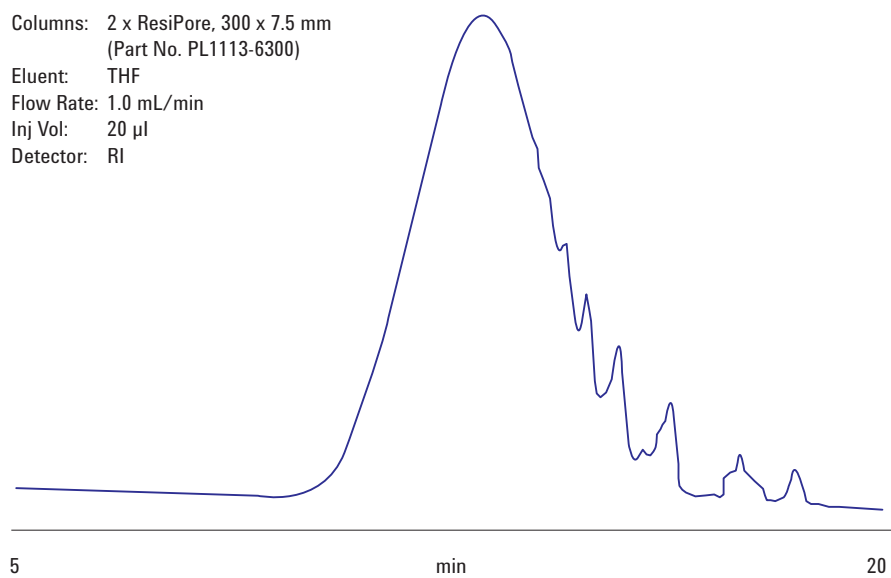


Figure 8. Chromatogram of higher molecular weight epoxy resin sample with some oligomer content

Columns: 2 x ResiPore, 300 x 7.5 mm
(Part No. PL1113-6300)

Eluent: THF

Flow Rate: 1.0 mL/min

Inj Vol: 20 µl

Detector: UV, 254 nm

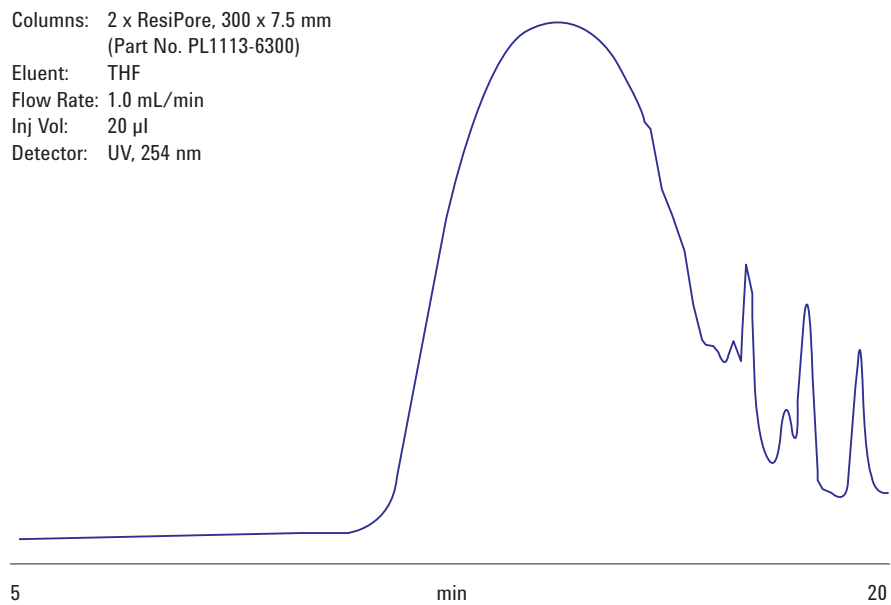


Figure 9. Chromatogram of an alkyd resin sample with a broad molecular weight distribution

Columns: 2 x ResiPore, 300 x 7.5 mm
(Part No. PL1113-6300)

Eluent: THF

Flow Rate: 1.0 mL/min

Inj Vol: 20 μ l

Detector: UV, 254 nm

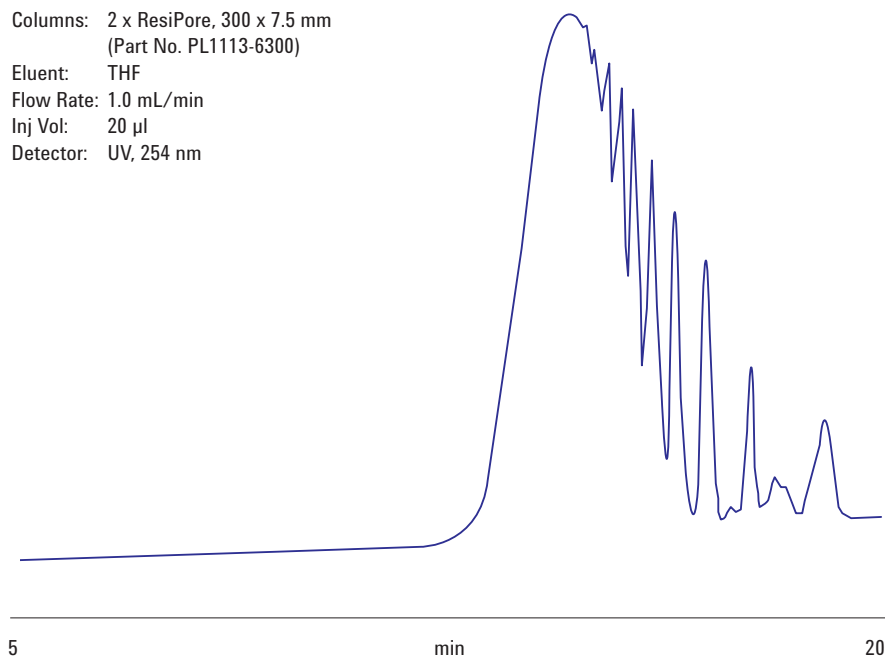


Figure 10. Chromatogram of a polyester sample showing oligomeric detail

Polyester polyol analysis

This separation demonstrates the resolution of the oligomeric species in a polyol sample prepared from adipic acid and butandiol using PLgel 3 μ m MIXED-E columns.

Columns: 2 x MesoPore, 300 x 7.5 mm
(Part No. PL1113-6325)

Eluent: THF

Flow Rate: 1.0 mL/min

Detector: UV, 254 nm

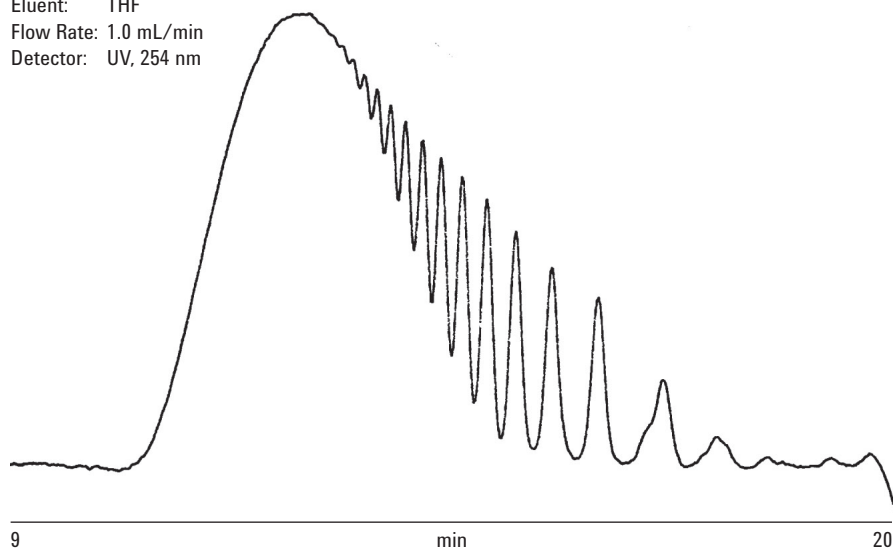


Figure 11. Chromatogram of a polyester polyol sample, a complex material with a UV chromophore

Phenolic resin analysis

Phenol-formaldehyde reactions produce two main products:

(a) Novolaks - under acidic conditions

(b) Resols - under basic conditions (excess aldehyde).

Chromatograms showing excellent oligomeric detail were produced for each product, when using high efficiency MesoPore columns.

Columns: 2 x MesoPore, 300 x 7.5 mm
(Part No. PL1113-6325)

Eluent: THF

Flow Rate: 1.0 mL/min

Detector: UV

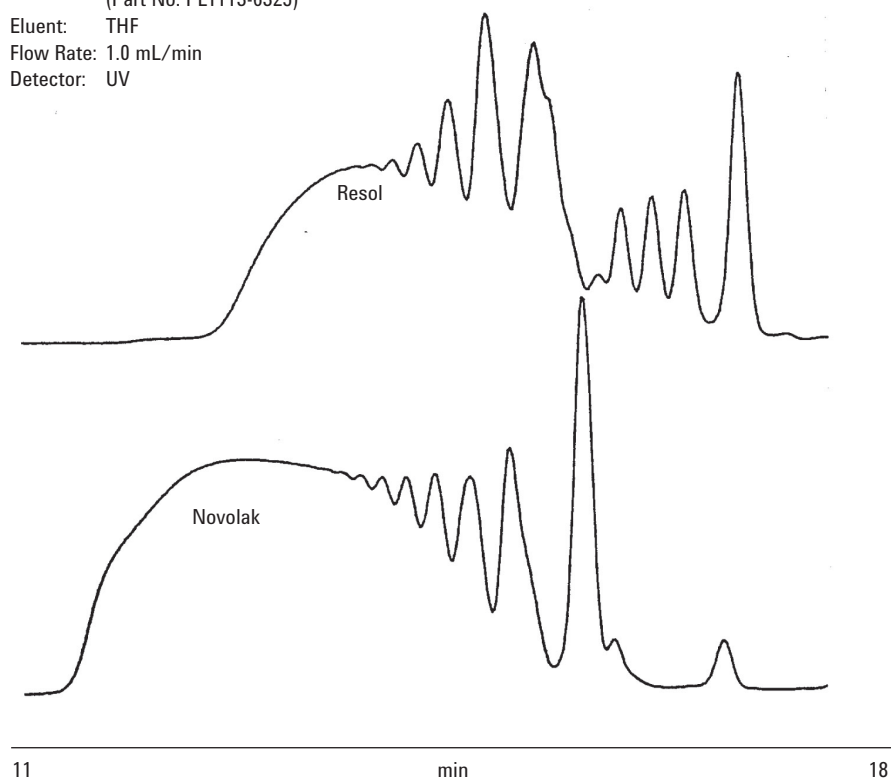


Figure 12. Overlaid chromatograms of two phenolic resin samples showing differences as a result of synthesis conditions

GPC analysis of adipate polyesters

Polyesters are produced from the condensation of a diacid with a dialcohol, eliminating water in the process. Depending on the acid and alcohol used, polyester can have a wide range of properties, including flexibility or hardness, stability to hydrolytic degradation and solvent, abrasion and shock resistance, properties which are useful for a wide range of applications. Adipate esters are produced by the condensation of a dialcohol with adipic acid.

Saturated adipate polyesters are used as cast elastomers; depending on the dialcohol used in the synthesis, linear or branched polyesters may be obtained. These polyesters are reacted with isocyanates to produce prepolymers with residual isocyanate groups that are precursors to mixed polyurethanes, a very important commercial class of material.

This application describes the analysis of two adipate polyesters by gel permeation chromatography (GPC) using two MesoPore columns in tetrahydrofuran with differential refractive index (RI) detection. Figure 13 shows two adipate polyester materials. The high resolution GPC columns are able to resolve the polymers into individual oligomers giving a characteristic peak shape that can be used to identify and 'fingerprint' different batches of polymers.

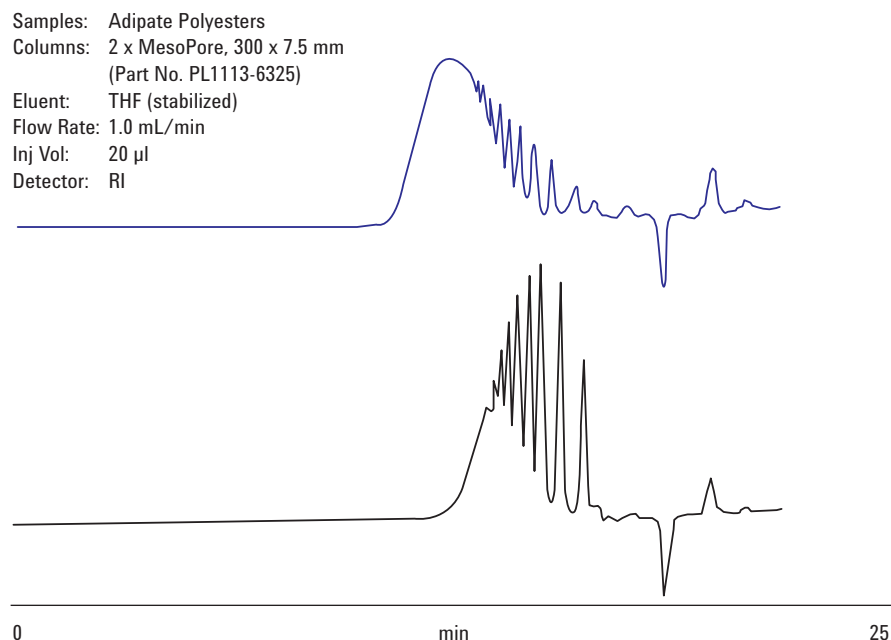


Figure 13. Overlaid chromatograms of adipate polyester samples showing clear differences between two batches

Higher molecular weight phenolic resins analysis

The term “phenolic resin” is used to describe a group of thermosetting resins produced through the reaction of phenol with an aldehyde. Phenolic resins were the earliest synthetic polymers to be developed (Bakelite, 1907), and possess useful mechanical and physical properties. Applications of phenolic resins include electrical insulation, molding, lamination and adhesives. Due to the relatively low cost and favorable properties of phenolic resins, they are produced in the greatest volume of all thermosetting polymers. Key characteristics of phenolic resins are their molecular weight distribution and oligomeric “fingerprint”, as these both have significant effect on the end use properties of the resin.

GPC is an ideal analytical tool for the examination of both of these characteristics. In this case, the use of high resolution GPC columns is advantageous, since these allow an optimized oligomeric separation and provide detailed information regarding the oligomeric sample composition.

These phenolic resins are non-polar so they can be analyzed in THF using PS/DVB columns.

In the GPC analysis detailed below, four distinct grades of phenolic resin have been analyzed by GPC using a ResiPore column set. Resulting from the small particle size (3 μm) and optimized pore size distribution of this column packing material, good resolution was obtained in the molecular weight range of interest.

The chromatography obtained from the GPC of each phenolic resin sample has been presented in Figure 14. Differential molecular weight distributions are given in Figure 15. This plot clearly shows significant differences in molecular weight distribution and the relative amounts of oligomeric material.

Columns: 2 x ResiPore, 300 x 7.5 mm
(Part No. PL1113-6300)
(conditioned with 10 injections of a
typical sample solution at 10 mg/mL)
Eluent: THF (stabilized with 250 ppm BHT)
Flow Rate: 1.0 mL/min
Inj Vol: 20 μm
Detector: RI

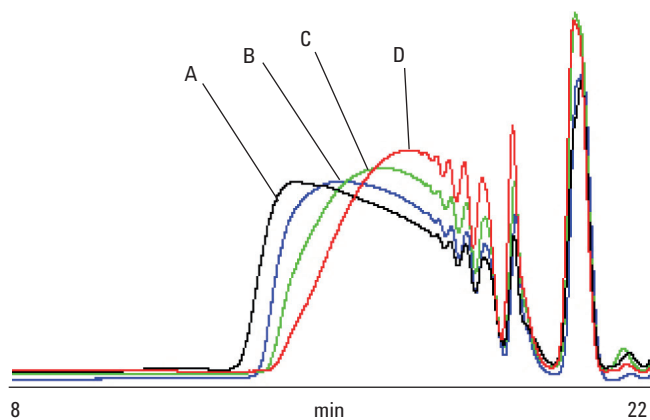


Figure 14. Overlaid GPC chromatograms obtained from four samples of phenolic resin showing differences in elution behavior

Columns: 2 x ResiPore, 300 x 7.5 mm
(Part No. PL1113-6300)
(conditioned with 10 injections of a
typical sample solution at 10 mg/mL)
Eluent: THF (stabilized with 250 ppm BHT)
Flow Rate: 1.0 mL/min
Inj Vol: 20 μm
Detector: RI

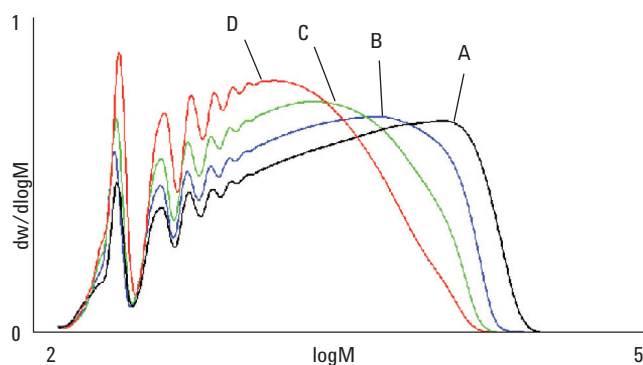


Figure 15. Overlaid molecular weight distributions from the GPC of four samples of phenolic resin - the same oligomers are present, but the overall molecular weights are very different

Preparative GPC separation of epoxy resin oligomers

Preparative GPC can be used to separate and isolate individual components of a sample based on size exclusion. By scaling up analytical separations, preparative GPC can be used to isolate practical quantities of individual components which can be used in further analysis. Agilent has developed the OligoPore preparative GPC column, which is ideally suited to the separation and isolation of individual oligomers from oligomer distributions and complex mixtures. This application illustrates the use of OligoPore preparative columns in the fractionation of epoxy oligomers. Figure 16 shows the general structure of an epoxy oligomer. A commercial epoxy resin, Epikote 828, is composed of two main epoxy oligomers where $n=0$ and $n=1$ and small amounts of the mono and di-epoxy water adducts.

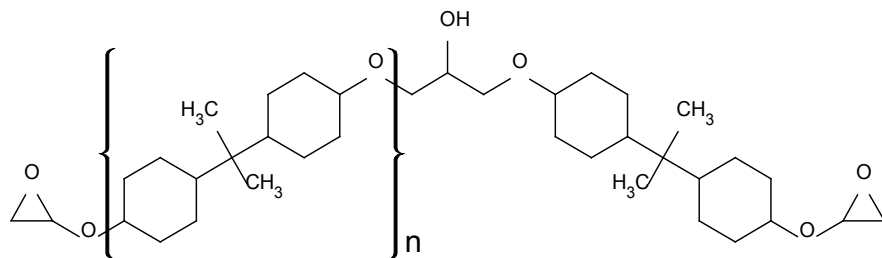


Figure 16. General structure of Epikote 828 epoxy resin oligomers

Analytical scale

Initially, the optimum loading of Epikote 828 on the OligoPore columns was analyzed on an analytical scale. Figure 17 shows analytical chromatograms at concentrations of 0.5% to 2.0% (w/v). The chromatograms show that Epikote 828 could be analyzed at a concentration of 2.0% (w/v) without serious loss of resolution.

Samples: Epikote 828, 0.5-2.0% (w/v)
Columns: 2 x OligoPore, 300 x 7.5 mm
(Part No. PL1113-6520)
Eluent: THF
Flow Rate: 1.0 mL/min
Inj Vol: 100 μ L
Detector: UV

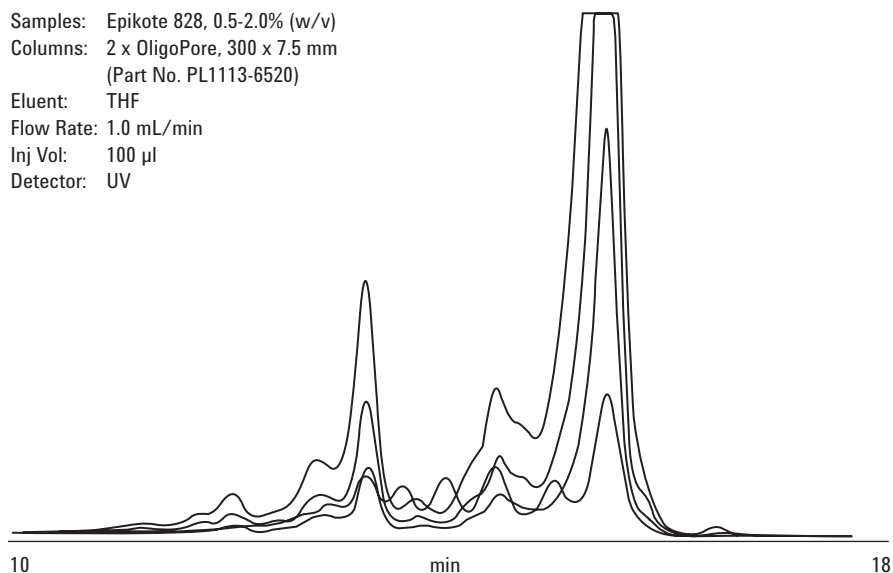


Figure 17. Overlaid chromatograms of epikote epoxy resin samples at different loadings

Preparative scale

OligoPore preparative columns were used to fractionate and purify the two oligomers from the resin. A preparative GPC system was set up with a 2 mL injection loop, two OligoPore 300 mm x 25 mm columns and a flow rate of 10.0 mL/min, an appropriate ten-fold scale-up over the analytical separation. The flow rate from the columns was split into two lines, ca 0.5 mL/min went to a UV detector, the remainder of the flow to a waste/fraction collector. The epoxy resin sample was injected at a concentration of 1.0% (w/v). Figure 18 shows a chromatogram of Epikote 828 obtained on the preparative columns indicating the resolution obtained. The sample was re-run and the two oligomers n=0 and n=1 were collected. The fractions were then analyzed on two OligoPore analytical columns.

Figure 19 shows the original analytical chromatogram of Epikote 828 run at a concentration of 2.0% (w/v) and an overlay of analytical chromatograms of the n=0 and n=1 oligomers collected from the OligoPore preparative GPC columns.

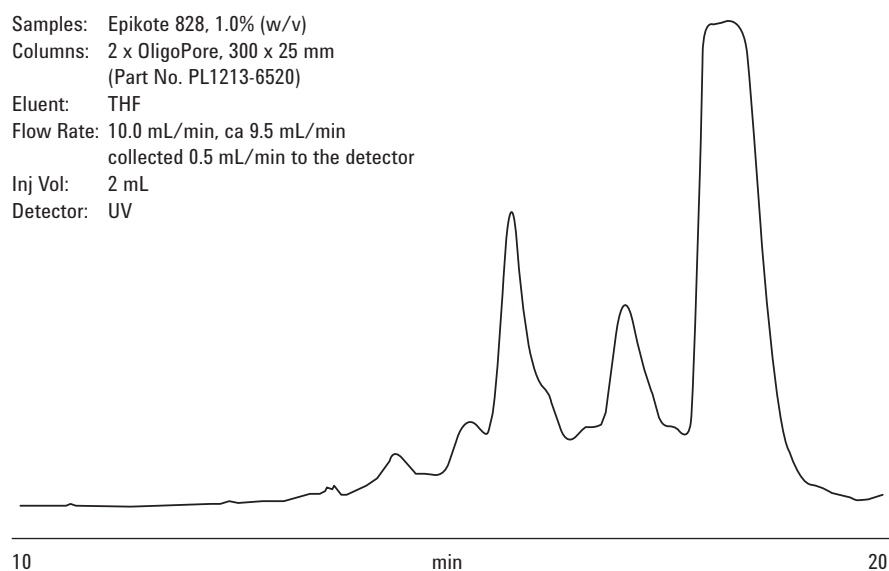


Figure 18. Chromatogram of an epikote 828 sample on a preparative scale column showing which oligomers could be isolated

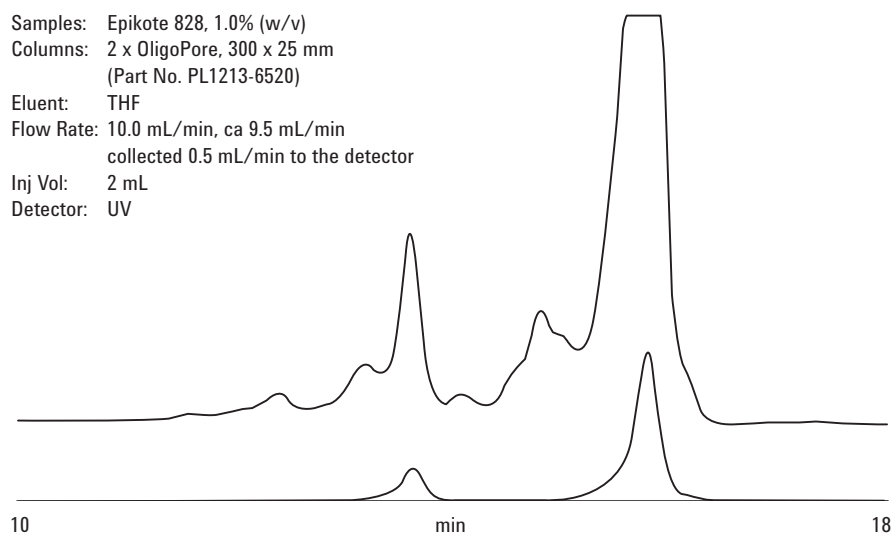


Figure 19. Overlaid chromatograms of an epikote 828 sample (top) and two separated fraction samples (bottom) showing the isolation of components through preparative GPC

Separation of epoxy resin oligomers

Epoxy resin prepolymers consist of oligomeric and polymeric diepoxides that are cured to form the finished product by the addition of a fixing or hardening agent. The formulation of the prepolymer is vital to controlling the physical properties of the final product. High resolution GPC can be used to investigate the oligomeric distributions of epoxy resin prepolymers for purposes of both formulation and quality control. When studying low molecular weight epoxy resins which elute close to total permeation, the Agilent evaporative light scattering detector (ELSD) is a good choice of detector due to the lack of system peaks, high sensitivity and excellent baseline stability obtained. This application outlines the analysis of four grades of epoxy resin using OligoPore columns and an ELS detector.

Figure 20 shows chromatograms of four different epoxy resins obtained on the OligoPore columns. The polymeric component of each of the samples was excluded on the OligoPore columns giving rise to a large peak at around 9 minutes, however, the oligomers were clearly resolved. The presence of peaks with identical retention times indicated that some of the same oligomers were present in each sample, however, clear differences in the oligomeric distributions of the four samples could be seen.

Samples: Epoxy resins
Columns: 2 x OligoPore, 300 x 7.5 mm
(Part No. PL1113-6520)
Eluent: THF + 250 ppm BHT
Flow Rate: 1.0 mL/min
Inj Vol: 100 µl
Detector: Agilent ELSD (neb = 40 °C,
evap = 80 °, gas = 1.0 SLM)

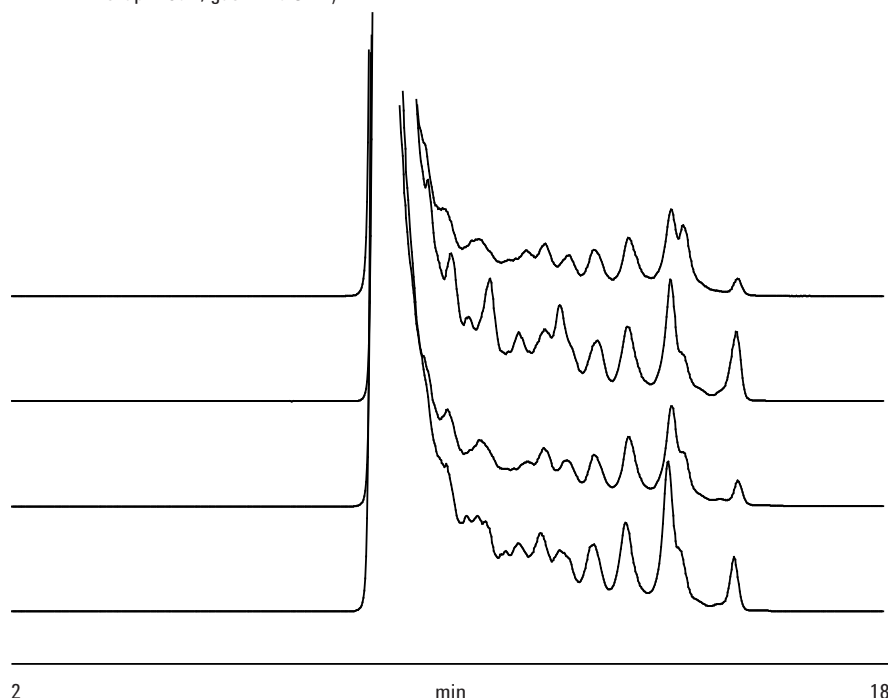


Figure 20. Overlaid chromatograms of epoxy resin samples, with excluded polymer components to focus on the oligomeric region

Characterization of high polarity resins

Increasingly, the choice of solvent for use as a GPC eluent is becoming more diverse since the polymers to be analyzed are more demanding in terms of solubility. Polar organic solvents are often the most suitable choice (see Table 1). However, such solvents usually exhibit relatively high viscosity, and demand the application of elevated temperature in order to improve the separation and reduce the column operating pressure.

The compatibility of GPC packing materials with this range of solvents assumes increasing importance in high performance separations of modern polymer systems. Column performance should be unaffected by solvent transfer, which demands a high degree of chemical and physical stability in the column bed.

Intermediate polarity packing

PolarGel columns contain macroporous copolymer beads with a surface of balanced polarity, comprising hydrophobic and hydrophilic components. As the polarity of the bead surface is intermediate between the non-polar PLgel and the highly polar PL aquagel-OH materials, PolarGel is ideal for the analysis of high polarity polymers that are insoluble in water yet show interaction effects with styrene/divinyl benzene columns due to their high polarity.

PolarGel columns perform well in many applications that do not work well on typical organic GPC columns. The low swell of the PolarGel material in a range of solvents explains the stability of the packing, see Figure 21.

Table 1. Solubility and eluent choices for different polymer types

Polymer Type	Solvent
ABS	DMF
Cellulose	DMSO/DMAC
Poly(acrylates)	DMF/DMAC
Poly(acrylonitrile)	DMF
Poly(ethylene oxide)	DMF
Poly(urethane)	DMF/DMAC
Poly(vinyl pyrrolidone)	DMF/DMAC

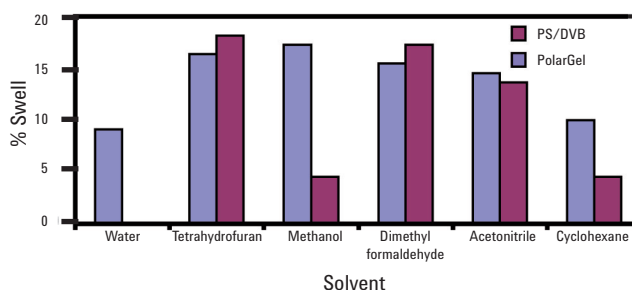
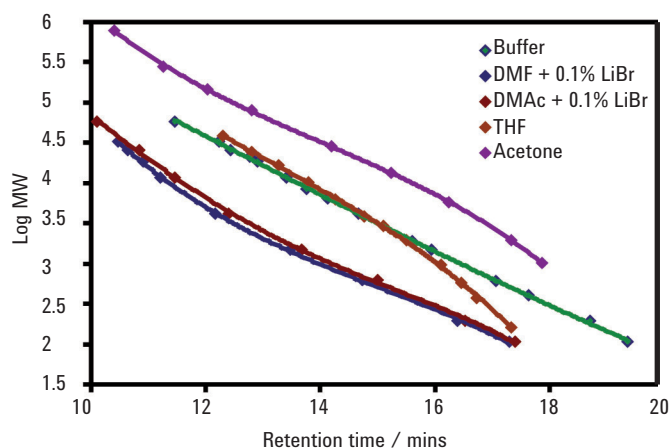


Figure 21. Swell data for PolarGel and a PS/DVB material showing the low swell of PolarGel

Broad solvent compatibility

As PolarGel columns combine intermediate surface polarity with low swell and high mechanical stability, they are used in a wide range of highly polar solvents, such as water, dimethyl formamide (DMF) and dimethyl acetamide (DMAc), and relatively low polarity solvents, such as tetrahydrofuran (THF), see figure 22. PolarGel-L columns are MIXED bed columns containing a number of constituents carefully blended to give a wide operating range focused at low molecular weight, making them suitable for a variety of applications up to ca. 30,000 g/mol (polyethylene glycol/oxide in water).



Samples: EasiVial PEG/PEO and PMMA
Standards (4 mL vials)
Columns: 2 x PolarGel-L, 300 x 7.5 mm
(Part No. PL1117-6830)
Eluent: Water, Acetone, DMF, DMAc, THF
Flow Rate: 1.0 mL/min
Inj Vol: 100 µl
Detector: Agilent PL-GPC 50 (RID)

Figure 22. Calibration curves of PolarGel-L in different solvents showing the near linear calibrations obtained in a solvent with a wide range of polarities

Analysis of phenol-formaldehyde Resins by GPC and PolarGel-M

Phenol-formaldehyde (P-F) resins are thermoplastic materials made with an excess of phenol in an acid catalyzed reaction with formaldehyde. P-F resins are commonly used as precursors to varnishes and other surface finish products.

PolarGel-M GPC columns are packed with low swell, macroporous copolymer beads that have a surface of balanced polarity, comprising hydrophobic and hydrophilic components. These allow PolarGel-M to be used in the analysis of high polarity polymers that are insoluble in water to give a more accurate representation of the molecular weight distribution of the polymer. If these polar polymers were to be analyzed with traditional styrene/divinylbenzene columns, interactions would cause artifacts in the peak shape and longer retention times, which would translate into apparently much lower molecular weight averages.

Two types of phenol-formaldehyde resin were analyzed to obtain an indication of differences in molecular weight, if any. The samples were made up in 0.2% (w/v) DMF, with 0.1% LiBr added to reduce sample aggregation, and injected without further treatment.

The results of the analyses are shown in the overlaid chromatograms and molecular weight distributions.

Columns: 2 x PolarGel-M, 300 x 7.5 mm
(Part No. PL1117-6800)
Eluent: DMF & 0.1% LiBr
Flow Rate: 1.0 mL/min
Inj Vol: 100 μ l
Temp: 50 $^{\circ}$ C
Detectors: Agilent PL-GPC 50 Integrated
GPC/SEC System, RI

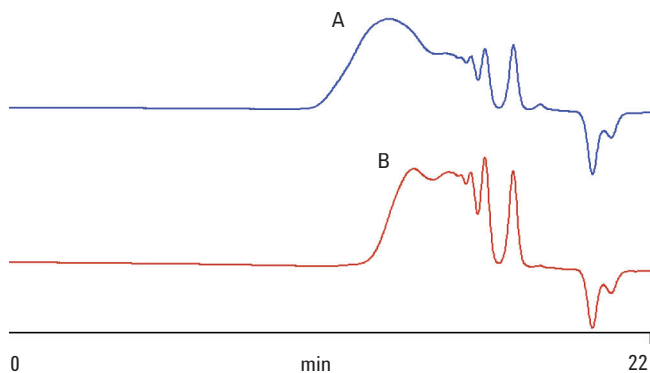


Figure 23. PolarGel-M reveals the composition of two phenol-formaldehyde resins, with clear differences between the materials

Columns: 2 x PolarGel-M, 300 x 7.5 mm
(Part No. PL1117-6800)
Eluent: DMF & 0.1% LiBr
Flow Rate: 1.0 mL/min
Inj Vol: 100 μ l
Temp: 50 $^{\circ}$ C
Detectors: Agilent PL-GPC 50 Integrated
GPC/SEC System, RI

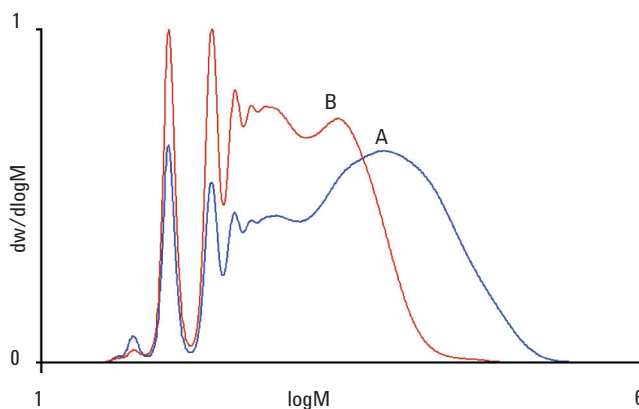


Figure 24. Overlaid molecular weight distributions of two phenol-formaldehyde resins show the same oligomers are present

GPC with PolarGel-M columns allows for the artifact-free calculation of the composition and molecular weight distributions of phenol-formaldehyde resins that are difficult to analyze on traditional, organic (PS/DVB) GPC columns.

GPC and PolarGel-M columns for the true representation of novolac resins

Novolac resins are thermoplastic materials made with an excess of phenol in an acid catalyzed reaction with formaldehyde. Novolacs are commonly employed as photoresists (light-sensitive materials used to form patterned surface coatings) and in varnishes. They have higher heat distortion temperatures and tend to be more expensive than regular epoxy resins.

PolarGel-M GPC columns are packed with low swell, macroporous copolymer beads that have a surface of balanced polarity, comprising hydrophobic and hydrophilic components. These allow PolarGel-M to be used in the analysis of high polarity polymers that are insoluble in water to give a more accurate representation of the molecular weight distribution of the polymer. If these polar polymers were to be analyzed with traditional styrene/divinylbenzene columns, interactions would cause artifacts in the peak shape and longer retention times, which would translate into apparently much lower molecular weight averages.

Two novolac resins were analyzed to obtain an indication of differences in molecular weight, if any. The samples were made up at 0.2% (w/v) in DMSO, with 0.1% LiBr added to reduce sample aggregation, and injected without further treatment.

Figure 25 shows the overlaid molecular weight distributions of two novolac resins.

GPC with PolarGel-M columns allows for the artifact, interaction free calculation of the composition and molecular weight distributions of novolac resins that are difficult to analyze on traditional, organic PS/DVB) GPC columns.

Columns: 2 x PolarGel-M, 300 x 7.5 mm
(Part No. PL1117-6800)
Eluent: DMSO & 0.1% LiBr
Flow Rate: 1.0 mL/min
Inj Vol: 50 $^{\circ}$ C
Detectors: Agilent PL-GPC 50 Integrated
GPC/SEC System, RI

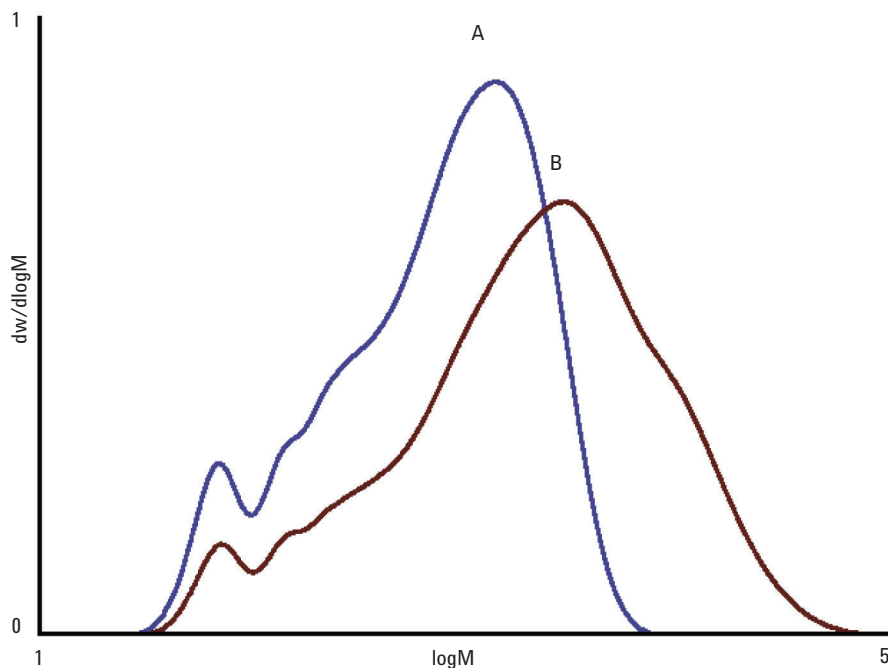


Figure 25. Overlaid molecular weight distributions of two novolac resins with very different molecular weights

Analysis of melamine resins by conventional Gel Permeation Chromatography using PolarGel-L columns on the PL-GPC 50

Melamine resins are durable thermosetting plastics formed by the condensation polymerization of melamine with formaldehyde. They are commonplace in the home as they are employed to laminate chipboard, creating inexpensive furniture, as well as being used in the manufacturing of kitchen tableware and food packaging. The molecular weight distribution of melamine resins determines many of the final properties of the polymer and therefore their application. Subtle differences in the molecular weight distribution of melamine resin samples is essential.

The molecular weight distributions of two different samples of melamine were investigated by conventional gel permeation chromatography using a set of two PolarGel-L (300 x 7.5 mm) columns and the PL-GPC 50 Integrated GPC/SEC System. The samples were analyzed in the polar organic solvent dimethylacetamide (DMAc) which contained 0.1% LiBr.

Sample: Two samples of melamine resin
Column: 2 x PolarGel-L, 300 x 7.5 mm
(Part No. PL1117-6830)
Eluent: DMSO & 0.1% LiBr
Flow Rate: 1.0 mL/min
Inj Vol: 50 °C
Detectors: Agilent PL-GPC 50 Integrated
GPC/SEC System, RI

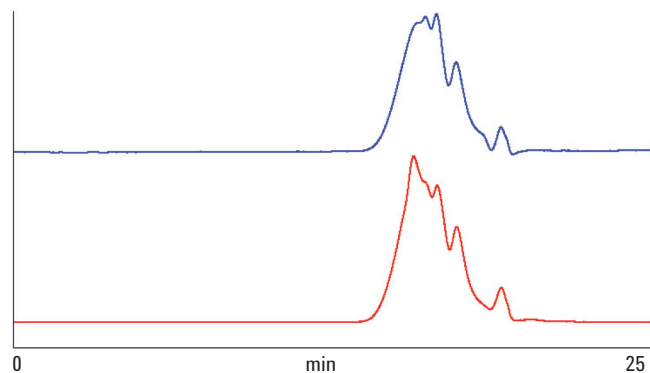


Figure 26. Chromatograms for two melamine samples showing obvious differences

Sample: Two samples of melamine resin
Column: 2 x PolarGel-L, 300 x 7.5 mm
(Part No. PL1117-6830)
Eluent: DMSO & 0.1% LiBr
Flow Rate: 1.0 mL/min
Inj Vol: 50 °C
Detectors: Agilent PL-GPC 50 Integrated
GPC/SEC System, RI

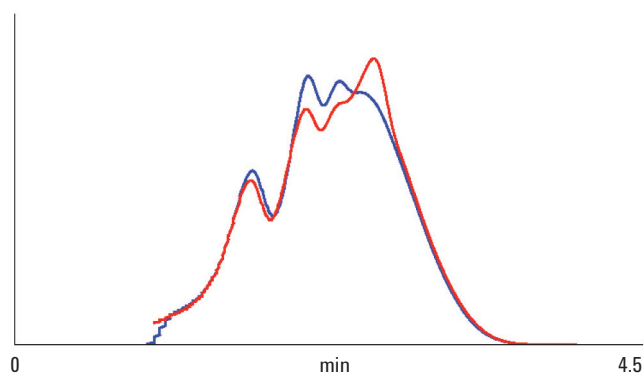


Figure 27. Overlaid molecular weight distributions for two melamine resin samples highlighting changes in the distributions

The two samples of melamine resins analyzed by conventional gel permeation chromatography on the PL-GPC 50 clearly had quite different molecular weight distributions, with differing ratios of the various oligomers present.

The PL-GPC 50 fitted with two PolarGel-L columns was used to successfully analyze two samples of melamine resin, indicating clear differences between the samples. The PolarGel-L columns are well suited to operation in highly polar solvents.

Analysis of resins

Resins comprise a complex group of natural and man-made, solid and semi-solid polymers for which there is no single, paramount analytical technique. To overcome this difficulty, Agilent makes a range of products in chromatography, NMR and spectroscopy that can be used for the investigation of this type of compound. Agilent's instruments and consumables elucidate the characteristics and composition of resins.

GC

For screening or fingerprinting of synthetic resins Agilent offers a range of high temperature GC columns, including DB-5ht, FactorFour VF-5ht, DB-2887, and CP-SimDist Ultimet. These columns are ideal for resin analysis using headspace sampling. To maximize column lifetime at high temperature, we recommend Agilent Gas Clean Filters to minimize oxygen and moisture content in carrier gas.

GC/MS

Threats to food safety through spoilage or adulteration have been of concern for many years. More recently, attention has focused on the perceived risk of harm from semi-volatile compounds given off by the plastic in which so much of our food is wrapped. These outgassed compounds, such as phthalates, can be detected by headspace GC/MS with the VF-5ms or VF-624ms columns and Agilent's ion trap instruments. Plastic wrapping of food can, however, have benefits when the atmosphere inside the pack is modified, by reducing the oxygen content to improve the keeping qualities of the food. Monitoring oxygen content is straightforward with CP-Molsieve columns. To assess CO₂ and moisture try Agilent's CP-PoraPLOT columns.

LC/MS

Additives are widely used to improve the performance characteristics of polymer resins. The analytical needs for additives analysis are qualitative identification, screening for potential contaminants (non-target analysis), and reliable, accurate quantitative determination of additive concentration in a complex matrix. It is a considerable analytical challenge to provide all of this information in a single analytical run. The Agilent 500-MS LC Ion Trap is ideal for this type of analysis, where sensitivity, reliability and productivity are essential. The 500-MS reliably detects and quantitates additives and non-target contaminants in complex matrices.

FT-IR

For most types of natural and synthetic resins, FT-IR from Agilent is a valuable tool for chemical identification of both known and unknown samples and also for QA/QC during manufacturing and post production. It can be used for the analysis of the liquid components (when they are in a viscous state) and of the final solidified samples. Depending on the type of resin, one can even study the process of resin-hardening (curing under different conditions) in 'real-time' using ATR-FT-IR. The fast scanning Agilent 670-IR FT-IR spectrometer is ideal for studying these kinetic processes. For most 'basic' resin analyses we recommend a 640-IR or 660-IR with a PIKE Diamond MIRacle ATR, for easy sample prep with a wide range of solvents and pH.

HPLC

PLRP-S 300Å columns provide oligomeric fingerprints of epoxy resins by reversed phase HPLC. Use these columns with any instrument in the Agilent 1200 Infinity Series, a suite of fully integrated, pre-configured and pre-tested HPLC instruments. Designed to get you quickly up and running, the Agilent 1200 Infinity Series is easy to customize, install and use.

NMR

If you work with compounds that are physically heterogeneous, such as non-filterable solid phase synthesis resins, then NMR with Agilent's NanoProbe is the best option. The NanoProbe combines the "high resolution" aspects of a liquids probe with the "MAS" aspects of a solids probe. Use NanoProbe with the new Agilent NMR System, engineered with a new technology designed for unsurpassed flexibility.

UV-Vis

UV-Vis spectroscopy is a valuable tool for the analysis of many polymers, including curable and composite dental resins. Agilent's Cary range of UV-Vis-NIR instruments has become the standard for researchers wanting to extend the boundaries of spectrophotometric measurement techniques. The range is equally at home in routine laboratories where reliability and ease of use are vital.

Find out how to take your resin analysis to the next level

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