

Application compendium

ANALYSIS OF FOOD BY GPC/SEC



Agilent Technologies

Analysis of food by GPC/SEC

Application compendium



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Introduction

Polymeric compounds are found in all foods, either as natural constituents or as additives to improve taste, texture or appearance. Other polymers are used extensively in food processing and packaging.

Food analysts are also concerned with compounds that should not be found in foods, such as pesticides and adulterants, and with the breakdown of foods as they are used in other processes. The composition of frying fat is a case in point.

One of the largest uses of polymers is in packaging. Polyolefins such as polyethylene are employed to make films and molded containers. Properties such as mechanical strength and toughness are vital in such end-use applications.

Gel permeation chromatography (GPC) is a versatile and valuable technique for all types of food analysis. What's more, although most GPC food analysis is carried out using water eluents, organic methods are also useful for more complex compounds, such as starch polysaccharides, and packaging polymers like polypropylene that are derived from crude oil. The purity of frying fats can also be assessed by GPC in organic eluents.

Pesticide levels must be closely monitored to ensure food is safe to eat. GPC can be used to separate and isolate individual components of a sample based on size exclusion, and can isolate toxic pesticides from contaminated food oils. By scaling up these analytical separations to preparative GPC, it is possible to isolate pesticide residues in sufficient quantity for further analysis or identification.

Many GPC food analyses benefit from the use of triple detection, because it combines concentration, viscometry, and light scattering detectors to assess the molecular weight distribution and molecular structure of polymers, without having to rely on column calibrations. This can be important when analyzing complex food polymers for which no structurally similar standards are available.

This compendium draws together many GPC techniques for the broader analysis of foods, including investigations of raw food materials, food additives, breakdown products, pesticide residues, and characterization of polymers used for food packaging. The versatility of gel permeation chromatography is ably demonstrated in food analysis.



Food additives

In food processing, formulation involves mixing active ingredients with one or more additives to increase the efficiency of the manufacturing process and improve the desirability of the food to consumers. For example, enhancements can preserve flavor or improve taste and appearance.

Other additives are used to modify acidity, mouth feel, and prolong shelf life. Additives that assist in manufacturing include anti-caking agents and flow improvers. It is very important for food manufacturers that their products are consistent in taste, appearance and quality, and all these parameters can be affected by additives. Because many food additives are polymeric in structure they are best analyzed by gel permeation chromatography or size exclusion chromatography, which provide a thorough understanding of the characteristics of polymer additives.



Pectins

Pectin is composed of a variety of complex heteropolysaccharides found naturally in fruits, such as apples, plums, grapes and cranberries (Figure 1). Structurally complex, pectins consist of 'smooth' and 'hairy' regions. The smooth regions are linear, partially methylated poly(D-galacturonic) acid, the hairy regions comprise alternating L-rhamnosyl and D-galacturonosyl residues containing L-arabinose and D-galactose branch points up to 20 residues long. As a result of this heterogeneous nature, pectins adopt complex structures in solution. Applications of pectin are related to the formulation of cross-links through hydrogen bonding of the carboxylic acid groups, and include use as gelling agents, thickeners and water binders.

A sample of pectin was analyzed on the Agilent PL-GPC 50 Integrated GPC/SEC System. The instrument was operated at 50 °C and incorporated a refractive index detector, an Agilent PL-GPC 50 viscometer and an Agilent PL-GPC 50 light scattering detector (collecting scattered light at 15° and 90°).

Two Agilent PL aquagel-OH MIXED-H 8 μm columns were used for the analysis. These high performance columns offer excellent resolution over a very wide range of molecular weights, simplifying column selection and providing a versatile analytical system. The sample was prepared accurately in the eluent and filtered before injection through a 0.45 μm disposable filter. For the purpose of light scattering calculations, an average dn/dc value was used for the sample.

Figure 2 shows the triple-detector chromatograms for the pectin sample. The chromatograms from the refractive index and light scattering detectors were clearly multimodal, as expected for a structurally heterogeneous material. Figure 3 is the calculated molecular weight distribution. From the viscometry and light scattering data, Mark-Houwink (log intrinsic viscosity versus log M) and conformation (log radius of gyration versus log M) plots were generated for the pectin, shown overlaid in Figure 4.

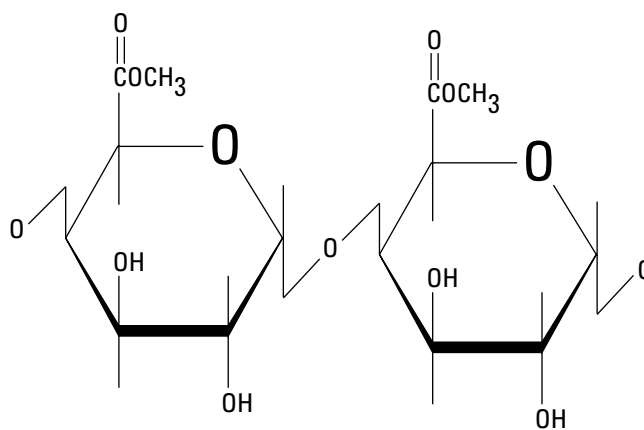


Figure 1. General structure of a pectin.

Conditions (Figures 2 to 4)

Columns:	2 x Agilent PL aquagel-OH MIXED-H 8 μm, 7.5 x 300 mm (p/n PL1149-6800)
Calibrants:	Agilent EasiVial PEO, 0.1 to 0.5 mg/mL
Sample:	Pectin
Sample concentration:	2 mg/mL in the eluent
Eluent:	0.2 M NaNO ₃ + 0.01 M NaH ₂ PO ₄ adjusted to pH 7
Injection volume:	200 μL
Flow rate:	1.0 mL/min
Temperature:	50 °C
Instrument:	Agilent PL-GPC 50 Integrated GPC/SEC System with DRI, viscometer, light scattering detector

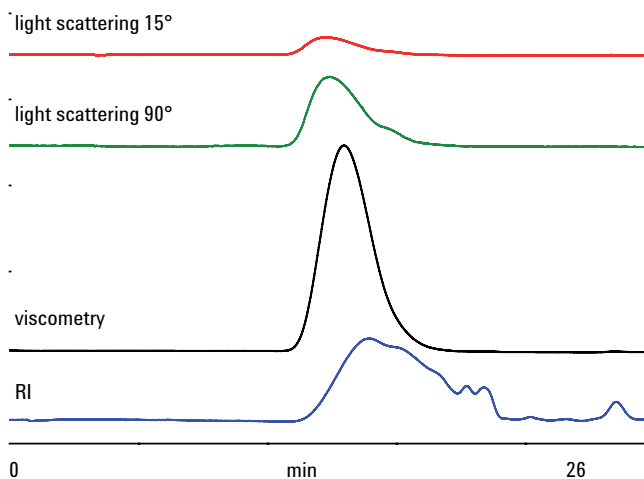


Figure 2. Triple-detector chromatograms of pectin (autoscaled) analyzed by the Agilent PL-GPC 50 with an Agilent PL aquagel-OH MIXED-H two-column set.

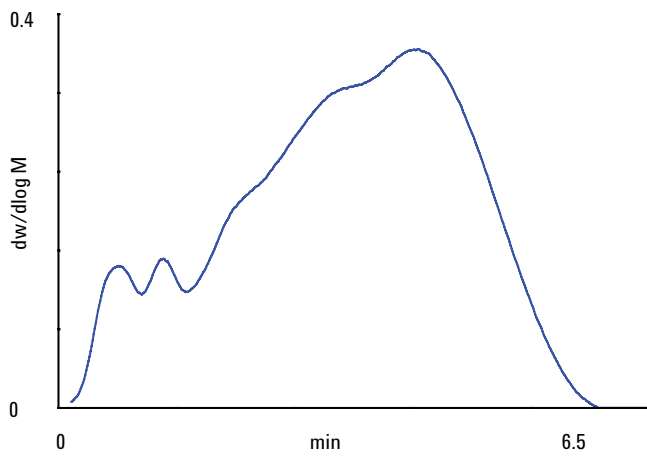


Figure 3. Molecular weight distribution plot calculated for pectin.

The Mark-Houwink, and to some extent the conformation plots, show curvature over the entire molecular density as a function of molecular weight, resulting from a variation in the relative amounts of 'smooth' and 'hairy' regions.

The PL-GPC 50 is a high resolution, cost effective integrated GPC system designed for operation from ambient to 50 °C. When coupled with PL aquagel-OH MIXED-H 8 µm columns, PL-GPC 50 viscometry and PL-GPC 50 light scattering detectors, the PL-GPC 50 makes maximum use of triple detection for the accurate determination of molecular weights of structurally complex and commercially important polymers. The wide resolving range of the columns allows complex natural materials such as pectin to be analyzed with confidence.

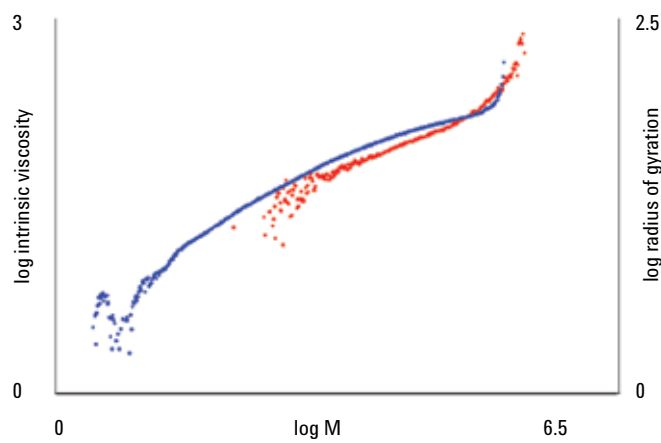
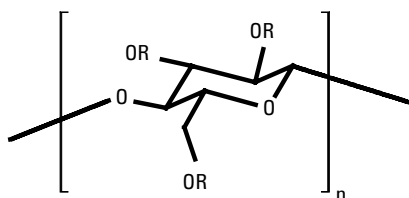


Figure 4. Overlaid Mark-Houwink and conformation plots for pectin.



Carboxymethyl cellulose

Carboxymethyl cellulose (CMC) is a derivative of cellulose with carboxymethyl groups (CH_2COOH) attached at some of the hydroxyl groups that typically make up the cellulose backbone. The general structure is shown in Figure 5. A sample of CMC was analyzed on the PL-GPC 50 Integrated GPC/SEC System. The instrument was operated at 50°C and incorporated a refractive index detector, and a PL-GPC 50 viscometer.



$\text{R} = \text{H}$ or $\text{CH}_2\text{CO}_2\text{H}$

Figure 5. General structure of the CMC monomeric repeat unit.

CMC has useful material properties such as high solution viscosity, which coupled with the low toxicity and nonallergenic nature of the material, results in its widespread use within the food science arena. Figure 6 shows the dual-detector chromatogram for the CMC sample. Figure 7 shows the molecular weight distribution calculated via Universal Calibration, a technique using the viscometer to determine molecular weights independent of the chemistry of the polymer calibrants employed. Figure 8 shows the Mark-Houwink plot generated from the viscometry data. For samples of CMC, the segregation of carboxy and hydroxyl functionalities can influence the size of the molecules in solution. In this case, the curvature of the Mark-Houwink plot can provide information about the structural or chemical homogeneity as a function of molecular weight.

Conditions (Figures 6 to 8)

Column: 2 x Agilent PL aquagel-OH 30 $8\ \mu\text{m}$, $7.5 \times 300\ \text{mm}$ (p/n PL1120-6830)
Calibrants: Agilent EasiVial PEG/PEO
Sample: Carboxymethyl cellulose
Sample concentration: $2.0\ \text{mg/L}$
Eluent: $0.2\ \text{M NaNO}_3$, $0.01\ \text{M NaH}_2\text{PO}_4$, pH 7
Injection volume: $100\ \mu\text{L}$
Flow rate: $1.0\ \text{mL/min}$
Temperature: Ambient
Instrument: Agilent PL-GPC 50 Integrated GPC/SEC System with DRI, viscometer

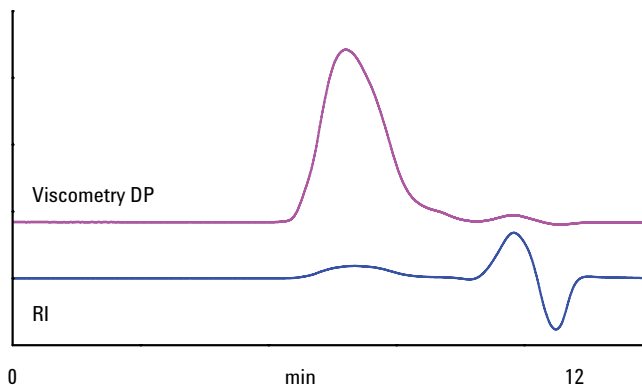


Figure 6. Refractive index/viscometry raw-data chromatograms obtained from a carboxymethyl cellulose sample analyzed on an Agilent PL-GPC 50 system with an Agilent PL aquagel-OH 30 two-column set.

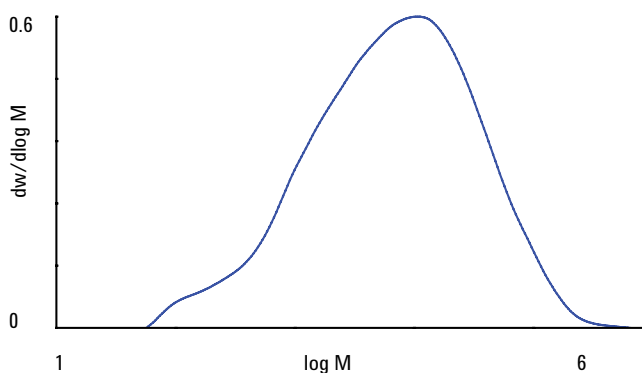


Figure 7. Molecular weight distributions obtained from a carboxymethyl cellulose.

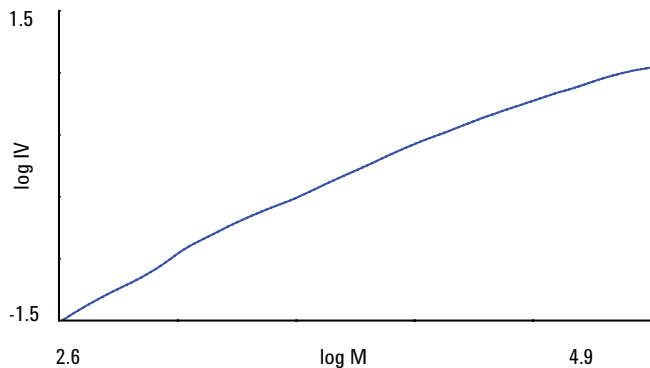


Figure 8. Mark-Houwink plot of a sample of carboxymethyl cellulose.

Starch

Starches are polysaccharides that contain glucose polymers amylose and amylopectin in a ratio of about 30:70, depending on the source. They have a great many industrial applications, with a very important role in the food industry. The source of the starch leads to different properties and therefore different end uses in foods. For example, corn starch is suited for confectionary products whereas potato starches are used in processed meats. They are also used as thickening agents in cooking. The molecular distribution and weight of the polymer determines many of its final properties therefore the end-use suitability for different applications.

Gel permeation chromatography with the Universal Calibration, employing a viscometer in combination with a differential refractive index detector, is used to determine accurate molecular weights for biopolymers, such as starches, that are independent of the standards used in the column calibration. Two starches were analyzed using these techniques. PLgel Olexis columns were chosen for the investigation because they are designed for the analysis of very high molecular weight polymers such as starches. The column resolves up to 100,000,000 g/mol (polystyrene in THF), and is packed with 13 μm particles to optimize efficiency and resolution without the risk of sample shear degradation during analysis.



Figure 9 shows a typical chromatogram of a starch by refractive index and viscometry. Figure 10 shows a comparison of two different starches and their molecular weight distributions, and Figure 11 the overlaid Mark-Houwink plots.

The two samples of starch analyzed by the Universal Calibration technique employing the Agilent 1260 Infinity Multi-Detector GPC/SEC System and PLgel Olexis columns showed stark differences in molecular weight distributions, with one of the samples having a bi-modal distribution. This accounted for the different thickening properties of the two materials.

The variations in the Mark-Houwink plots indicated that the materials were structurally very different, presumably due to the fact the samples were obtained from two separate sources.

Conditions (Figures 9 to 11)

Column: 3 x Agilent PLgel Olexis, 7.5 x 300 mm (p/n PL1110-6400)
 Sample: Starches
 Sample concentration: 2 mg/mL in the eluent
 Eluent: DMSO:DMAc (4:1) + 0.1% LiBr
 Flow rate: 1.0 mL/min
 Temperature: 60 °C
 Instrument: Agilent 1260 Infinity Multi-Detector GPC/SEC System, isocratic pump, thermostatted column compartment, manual injector, with DRI, viscometer

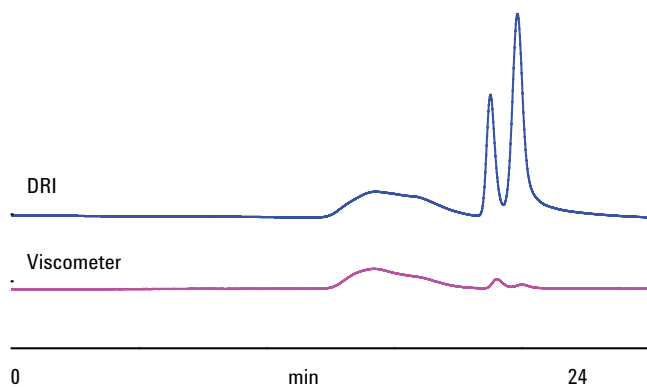


Figure 9. Viscometer and refractive index detection chromatograms for a starch on the Agilent 1260 Infinity Multi-Detector GPC/SEC System with an Agilent PLgel Olexis three-column set.

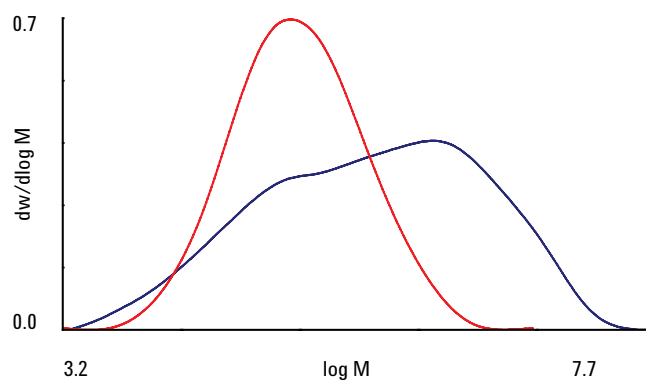


Figure 10. Overlaid molecular weight distributions (MWD) for two starch samples. Differences in MWD account for their different physical properties.

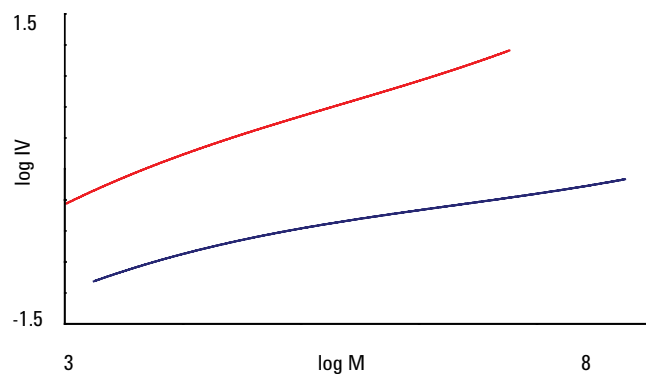


Figure 11. Overlaid Mark-Houwink plots for two starch samples reveals marked differences in their structure, with one of the samples having a much lower size in solution and therefore intrinsic viscosity than the other sample, which may be due to differences in the level of branching between the two materials.

Corn flour

Corn flour or cornstarch is the starch of the maize grain, ground from the endosperm of the corn kernel. Starches contain two structurally different polysaccharides, amylose and amylopectin. In corn flour, the ratio of these materials is typically around 25:75. When mixed with water, corn flour behaves as a non-Newtonian fluid showing typical shear thickening behavior, giving way to gentle pressure but resisting sudden impact. Many uses of corn flour in the food industry rely on the thickening and anticoagulant properties of the material.

Two samples of corn flour from different sources had displayed differing properties when used as thickening agents in a food application. It was thought that variations in levels of the linear amylose and the highly branched amylopectin polysaccharides were responsible for this behavior. To investigate the molecular structure of the materials, they were analyzed on an integrated GPC system. Molecular weight distributions were determined using the Universal Calibration method and structure of the samples was compared using the Mark-Houwink plot of log intrinsic viscosity as a function of log molecular weight. Increased amount of branched material would result in a contraction in the molecular size of the materials with a downward deviation in the Mark-Houwink plot.

The corn flours were analyzed by a PL-GPC 50 fitted with a PL-GPC 50 DRI and viscometer and PLgel 10 μm MIXED-B columns, which provide high resolution of polymers with high molecular weights even in demanding eluents.

Figure 12 shows a chromatogram of a sample of corn flour. Clear differences in the molecular weight distributions of the samples are apparent in Figure 13, and the effect of changes to the content of amylose and amylopectin could be observed in the shifts of the Mark-Houwink plots (Figure 14).

The results show that the two samples of corn flour have markedly different sizes in solution, indicative of structural differences between the materials. This is most likely caused by variations in the ratio of amylose to amylopectin in the two samples. As a result of these size differences, conventional GPC employing only a refractive index detector would give an anomalous result for the two samples, as size is used to derive the molecular weights calculated by conventional GPC.



Conditions (Figures 12 to 14)

Columns: 3 x Agilent PLgel 10 μ m MIXED-B, 7.5 x 300 mm
(p/n PL1110-6100)
Sample: Corn flour
Sample concentration: 2 mg/mL in the eluent
Eluent: Dimethyl sulfoxide + 0.1% LiBr
Flow rate: 1.0 mL/min
Temperature: 50 °C
Instrument: Agilent PL-GPC 50 Integrated GPC/SEC System with DRI, viscometer

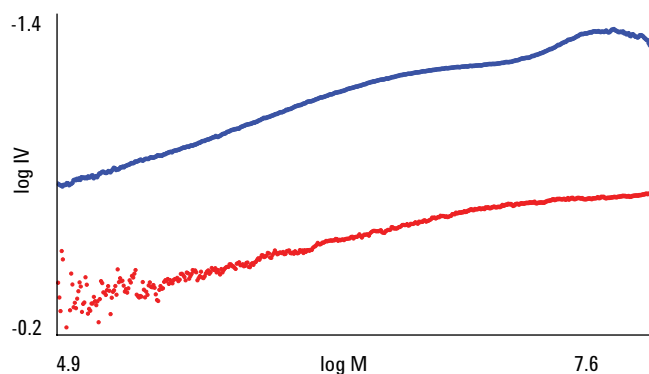


Figure 14. Overlaid Mark-Houwink plots for two samples of corn flour.

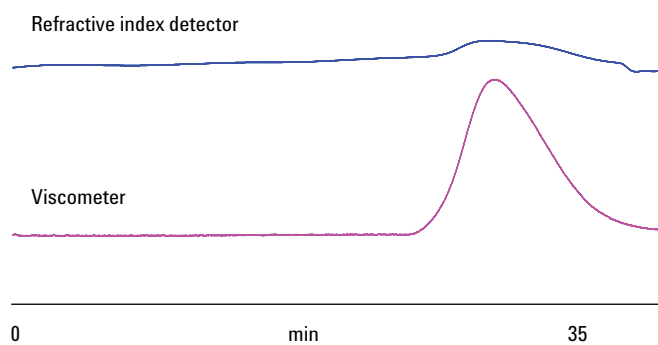


Figure 12. Example chromatograms of one of two corn flour samples produced by an Agilent PL-GPC 50 Integrated GPC/SEC System with Agilent PLgel 10 μ m MIXED-B columns.

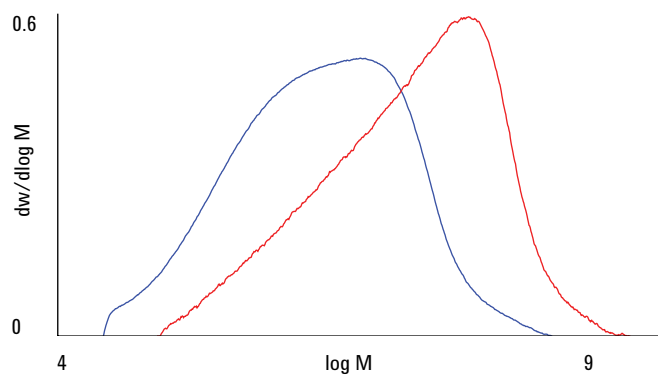


Figure 13. Overlaid molecular weight distributions for two corn flours.

Pullulan and dextran

Many polysaccharides show large structural differences due to the manner in which they are synthesized. This is most commonly seen in the presence of branches on the polymer chains of some polysaccharides, which strongly influences properties such as solution viscosity. Pullulan polysaccharide is composed of maltotriose units in the polymer backbone, produced from starch by the action of a fungus. Pullulan has a linear structure, whereas dextran, manufactured from sucrose by bacterial action, is a complex glucan with many differing components and a highly branched structure. Investigating the structure of polysaccharides is of interest for determining their properties in applications such as food additives.

Two samples of polysaccharide were analyzed by GPC viscometry; A pullulan with a linear structure, and a highly branched dextran.

Figure 15 shows an overlaid multidetector chromatogram for a sample of pullulan polysaccharide. The material eluted as a broad peak, with a small late eluting peak on the DRI detector due to solvent imbalances.

Figure 16 is an overlay of the accurate molecular weight distributions of the two samples under investigation. As can be seen, they have very different molecular weight distributions

Figure 17 shows the overlaid Mark-Houwink plot of log intrinsic viscosity as a function of molecular weight for the two samples. Compared to the pullulan, the dextran shows a marked shift of the Mark-Houwink plot to lower intrinsic viscosity values at any given molecular weight. This indicates that dextran is smaller in solution than pullulan across the molecular weight range, a result of the presence of branching on the dextran molecules. The dextran plot is complex and shows some changes in slope, indicating that the degree of branching varies across the range of molecular weight, as expected for a complex material, with the data indicating slightly more branching at lower molecular weight.

Conditions (Figures 15 to 17)

Columns:	2 x Agilent PL aquagel-OH MIXED-M 8 μ m, 7.5 x 300 mm (p/n PL1149-6801)
Sample:	Pullulan and dextran
Sample concentration:	2 mg/mL in the eluent
Eluent:	0.2 M NaNO ₃ + 0.01 M NaH ₂ PO ₄
Injection volume:	200 μ L
Flow rate:	1.0 mL/min
Temperature:	40 °C
Instrument:	Agilent 1260 Infinity Multi-Detector GPC/SEC System, isocratic pump, thermostatted column compartment, manual injector, with DRI, viscometer

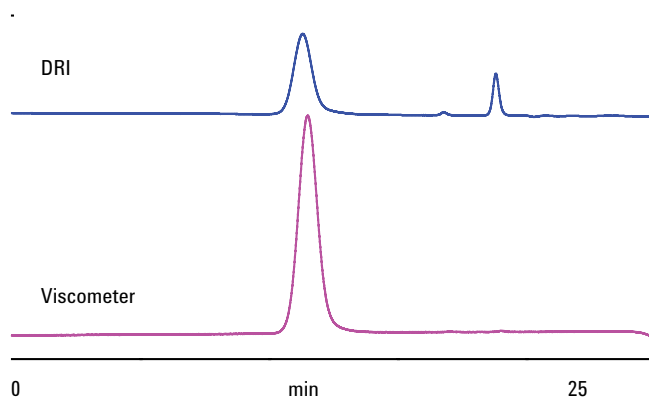


Figure 15. Overlaid multidetector chromatogram for a pullulan polysaccharide on the Agilent 1260 Infinity Multi-Detector GPC/SEC System with Agilent PL aquagel-OH MIXED-M columns.

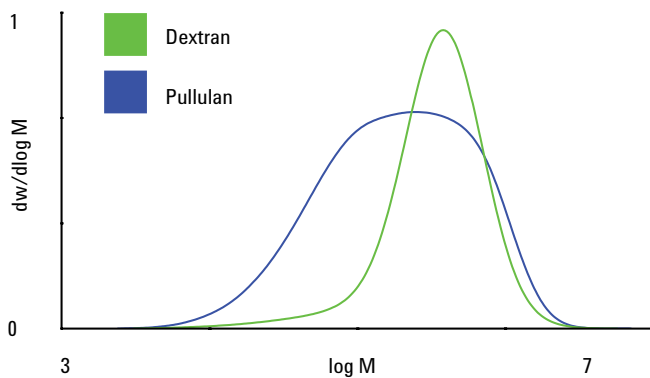


Figure 16. Overlaid multidetector molecular weight distributions of two samples of polysaccharide.

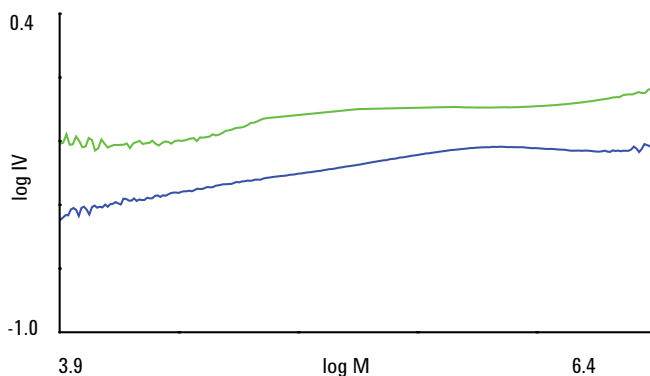


Figure 17. Overlaid Mark-Houwink plots for two polysaccharide samples.

The data illustrate how multidetector GPC, employing the Agilent 1260 Infinity Multi-Detector GPC/SEC System, can be used to clearly see structural differences between pullulan and dextran with a highly branched structure when coupled to a high resolution SEC column set capable of analyzing water-soluble polymers of quite high molecular weight.

Dextran can also be analyzed using single-detector GPC, as shown in Figure 18. In this analysis a dextran narrow polydispersity standard was used.

Conditions

Columns: 2 x Agilent PL aquagel-OH MIXED-M 8 μ m, 7.5 x 300 mm (p/n PL1149-6801)
 Sample: Dextran narrow standard
 Sample concentration: 2 mg/mL
 Eluent: 0.2 M NaNO₃, 0.01 M NaH₂PO₄, pH 7
 Injection volume: 200 μ L
 Flow rate: 1.0 mL/min
 Temperature: 40 °C
 Instrument: Agilent 1260 Infinity Multi-Detector GPC/SEC System, isocratic pump, thermostatted column compartment, manual injector, with DRI

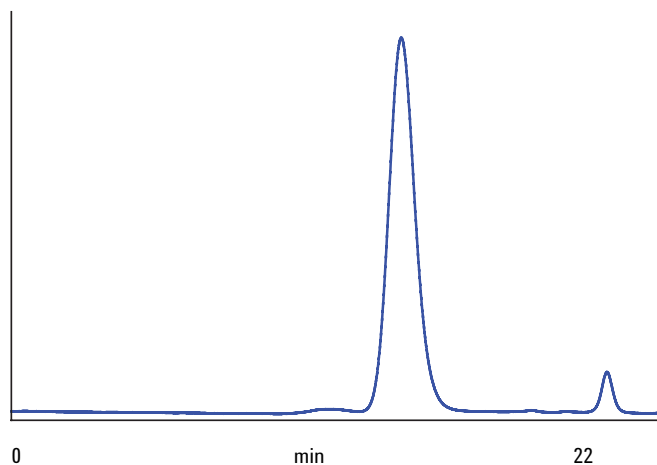


Figure 18. Typical chromatogram of a narrow polydispersity dextran on a two-column set of Agilent PL aquagel-OH MIXED-M columns.

Gelatin

Food-grade gelatins are biologically derived materials used in the food industry as thickening agents. SEC analysis of gelatins yields critical molecular weight information upon which the physical properties of the polymer (such as the setting properties) depend. Linear PL aquagel-OH MIXED-H 8 μm columns were used. These columns resolve up to 10,000,000 g/mol (polyethylene glycol/oxide equivalent), with excellent resolution over a very wide range of molecular weights, simplifying column selection and providing a versatile analytical system.

The eluent was prepared as a buffer with its pH adjusted by the addition of 0.1 M NaOH. The samples were accurately prepared as 1.0 mg/mL solutions in the eluent. The light scattering detector was first calibrated using a pullulan polysaccharide standard prepared at 1.0 mg/mL. From the known concentration, M_p and dn/dc of the calibrant, the detector constants and inter-detector volume for the system were calculated allowing molecular weight calculations to be performed.

From the RI chromatogram, dn/dc was calculated for the gelatin sample as the sample had been prepared at known concentration. This value of dn/dc was then used to calculate a bulk M_w value from the 90° and the 15° light scattering data.

The RI and light scattering data was also used to perform an SEC slice-by-slice molecular weight calculation for the gelatin sample using both LS signals. The bulk M_w values were 174,000 (90°), 189,850 (15°), and 184,800 (SEC)

Figure 19 shows the RI and the 90° and 15° light scattering data for the gelatin sample. Light scattering detection is more sensitive to higher molecular weight species, hence the 90° and 15° light scattering chromatograms placed more emphasis on high molecular weight material than the RI chromatogram. The RI chromatogram also contained a negative peak due to compositional differences between the sample, solvent and eluent, which was not observed by light scattering.

Conditions

Columns:	2 x Agilent PL aquagel-OH MIXED-H 8 μm , 7.5 x 300 mm (p/n PL1149-6800)
Sample:	Gelatin
Sample concentration:	2 mg/mL in the eluent
Eluent:	0.2 M NaNO ₃ + 0.01 M NaH ₂ PO ₄ at pH 7
Flow rate:	1.0 mL/min
Instrument:	Agilent 1260 Infinity Multi-Detector GPC/SEC System, isocratic pump, manual injector, with dual-angle light scattering detector, DRI

The wide molecular weight operating range of PL aquagel-OH MIXED-H 8 μm columns makes them particularly suited to the analysis of water soluble polymers with intermediate to high molecular weight. The use of a simple buffer solution as the eluent for the analysis of gelatins reduces interaction between the sample and the columns ensuring that good chromatography is obtained.

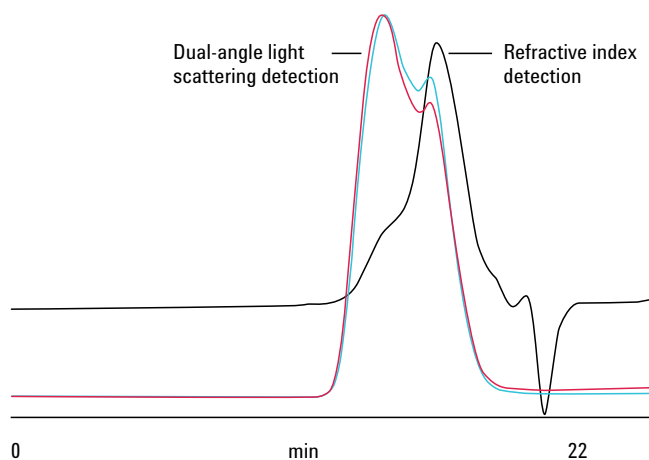


Figure 19. Refractive index, and 90° and 15° light scattering data for a gelatin sample on an Agilent 1260 Infinity Multi-Detector GPC/SEC System with an Agilent PL aquagel-OH MIXED-H two-column set.



Gums

Gums are complex polysaccharides used widely in the food industry as viscosity modifiers or gelling agents that provide characteristic shape and consistency to many foods. Most are derived from natural products, such as seaweed or locust bean. Others are extracted from microbial fermentation, or from animal tissue. Conversely, cellulose gum can be synthesized by reacting cellulose with chloroacetic acid. The physical properties and processibility of these water-soluble polymers are related to their molecular weight distribution, which can be determined by aqueous size exclusion chromatography.

Gum Arabic is a natural gum made from the hardened sap of acacia trees. It is produced across the African Sahel and the Middle East, and is an important ingredient in soft drinks and candies. The proportions of constituent polymers in gum Arabic vary widely. A comparison of the molecular weight distributions of 'good' and 'bad' samples shows clear differences between two batches of gum Arabic, the 'bad' sample having considerably greater high molecular weight material (Figure 20)

Conditions

Columns: 2 x Agilent PL aquagel-OH 60 8 μm , 7.5 x 300 mm (p/n PL1149-6860),
1 x PL aquagel-OH 40 8 μm , 7.5 x 300 mm (p/n PL1149-6840)
Sample: Gum Arabic
Sample concentration: 2 mg/mL in the eluent
Eluent: 0.2 M NaNO_3 , 0.01 M NaH_2PO_4 , pH 7
Flow rate: 1.0 mL/min
Instrument: Agilent 1260 Infinity Multi-Detector GPC/SEC System, isocratic pump, manual injector, with DRI

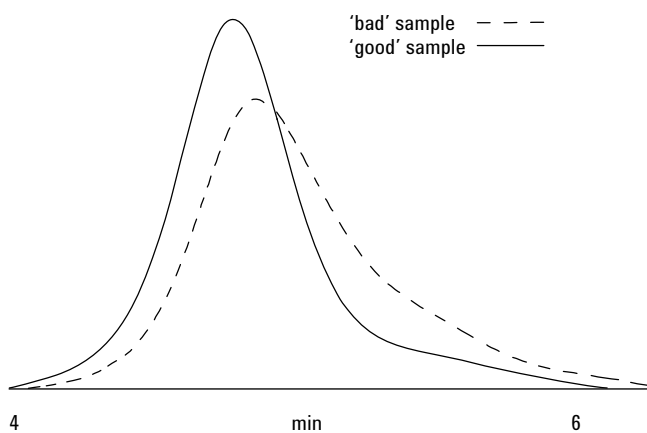


Figure 20. Overlay chromatogram showing molecular weight distribution of two batches of gum Arabic produced on an Agilent PL aquagel-OH three-column set.





Food flavorings

Many modern foodstuffs contain flavorings, which are added to enhance the taste of food and improve its desirability. Often complex mixtures of chemicals are required to give the specified flavor. Analyzing and controlling the relative amounts of these flavorings is a vital part of the quality control process to ensure product consistency.



A sample of flavoring that contained several low molecular weight maltodextrins and a high molecular weight starch acting as a carrier was analyzed by SEC. The performance of flavoring of this type depends upon the relative distribution of maltodextrin components within the sample. Therefore, for the analysis of the flavoring, columns were selected that focus on the resolution of the low molecular weight maltodextrin but exclude the less important starch from the analysis. Aqueous SEC is an excellent tool for characterizing flavorings, making use of PL aquagel-OH 30 8 μm columns in buffer at pH 7 using RI detection.

PL aquagel-OH columns operate across a wide range of eluent conditions for high performance analysis of analytes with neutral, ionic and hydrophobic moieties, singly or combined. Pullulan polysaccharide narrow standards were used to generate the calibration.

Figure 21 shows a chromatogram of the food additive. Some of the starch component was partially resolved at high molecular weight but the major proportion was excluded, giving rise to the sharp peak at the exclusion limit of the column set (at approximately nine minutes). The maltodextrin distribution of the flavoring was separated from the starch and resolved into three peaks with several smaller shoulders - the Mp values of the major components relative to polysaccharide are shown. Quantification of the relative peak heights of the individual maltodextrin components would allow quality control of the flavoring.

Conditions

Columns: 2 x Agilent PL aquagel-OH 30 8 μm , 7.5 x 300 mm (p/n PL1120-6830)
Sample: Maltodextrin flavorings in starch
Eluent: 0.2 M NaNO_3 + 0.01 M NaH_2PO_4 pH 7
Flow rate: 1.0 mL/min
Instrument: Agilent 1260 Infinity Multi-Detector GPC/SEC System, isocratic pump, manual injector, with DRI

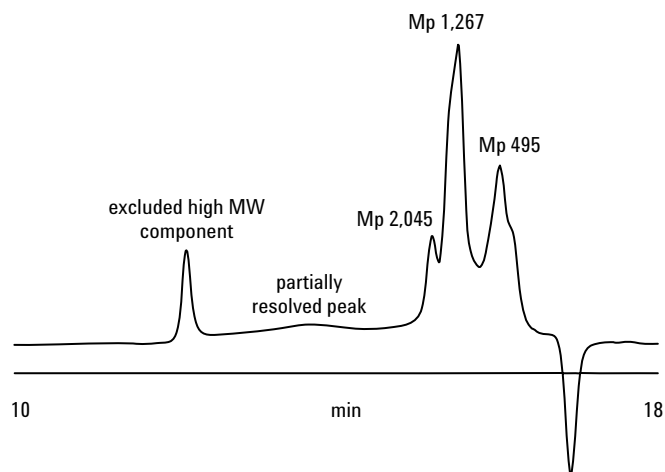


Figure 21. Raw data chromatogram of a food flavoring.

Using SEC with PL aquagel-OH columns allows investigators to focus on low molecular weight maltodextrins in the presence of high molecular weight starches, greatly facilitating quality control of these important food flavorings.





Frying fats

The purity of frying fats can be assessed by gel permeation chromatography in organic eluents.

The analysis involves a separation of the oligomeric glycerides based on molecular size in solution. It is possible to separate the major component (monoglyceride) from the minor components (diglyceride, triglyceride) of frying fat (Figure 22 and Table 1), and subsequently perform a quantitative analysis to obtain information relating to the purity of the monoglyceride.

Conditions

Columns: 2 x Agilent PLgel 5 μm 500Å, 7.5 x 300 mm (p/n PL1110-6525)
 Sample: Frying fat
 Sample concentration: 0.5%
 Eluent: THF (stabilized)
 Injection volume: 20 μL
 Flow rate: 1.0 mL/min
 Instrument: Agilent PL-GPC 50 Integrated GPC/SEC System with DRI

Table 1. Chromatographic characteristics of three frying fat glycerides.

Peak	Retention time (min)	Area (%)
1	12.97	5.3
2	13.55	11.3
3	14.68	83.4

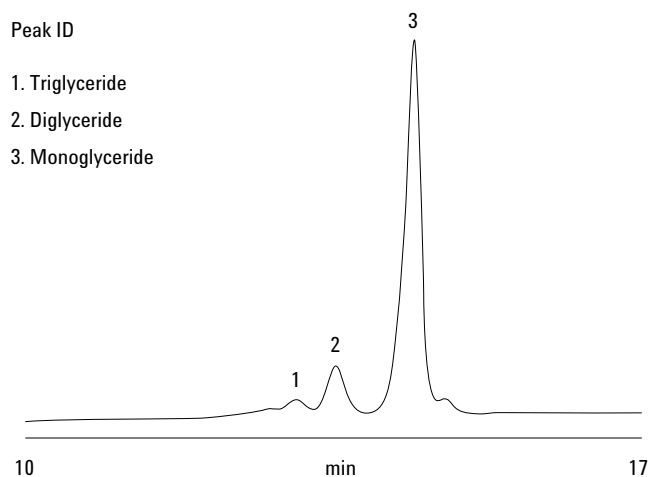


Figure 22. Three glycerides in a frying fat separated by Agilent PLgel 5 μm columns.

Gel permeation chromatography with PLgel columns can be used to determine the ratio of components in complex materials such as frying fats.

During the frying process, a wide variety of chemical reactions results in the formation of compounds with high molecular weight. The presence of air in the frying system and the heating of fats initiate a cycle of thermal and thermo-oxidative decomposition that results in the formation of polymeric compounds. The quality and properties of the fats are dependent on the levels of these higher molecular weight materials.

Typical separations of triglycerides from plant and animal sources are shown in Figures 23 and 24. For peak detection, a conventional differential refractometer can be used (as shown in Figure 23). As an alternative, an evaporative light scattering detector can provide improved sensitivity and baseline stability to aid quantification (see Figure 24). An outline of the method and its validation has been published in an IUPAC paper¹.

Conditions

Columns: 2 x Agilent PLgel 5 μm 100 \AA , 7.5 x 300 mm (p/n PL1110-6520)
 Eluent: THF
 Flow rate: 0.5 mL/min
 Instrument: Agilent PL-GPC 50 Integrated GPC/SEC System with DRI (Figure 22) or Agilent 1260 Infinity ELSD (Figure 23)

Peak ID

1. Polymer
2. Triglyceride
3. Diglyceride
4. Monoglyceride
5. Fatty acid

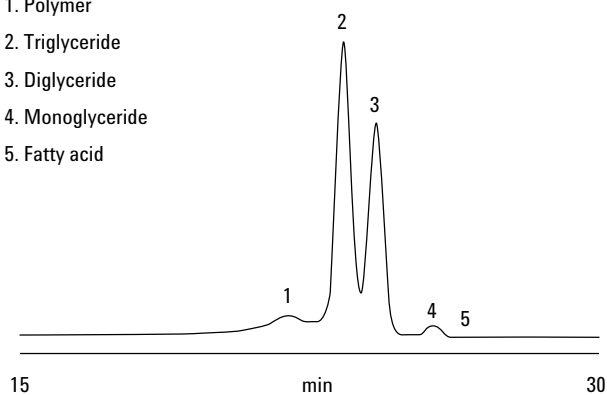


Figure 23. Fish oil analyzed on an Agilent PLgel 5 μm two-column set with refractive index detection.

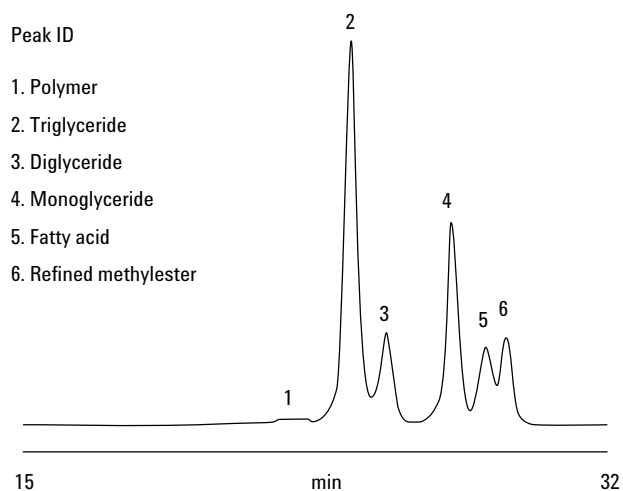


Figure 24. Grape seed oil separated on an Agilent PLgel 5 μm two-column set with evaporative light scattering detection to provide baseline stability and improved sensitivity compared to refractive index detection.

Gel permeation chromatography with PLgel 5 μm columns is an appropriate method for quantifying triglycerides in frying oils. If peak detection is the main requirement a conventional differential refractometer is perfectly adequate. However, for quantification an Agilent evaporative light scattering detector improves sensitivity and baseline stability.

Alkyl glycerides are discrete molecules commonly found in frying fats and oils. PLgel 3 μm 100 \AA columns have been specifically designed for the analysis of low molecular weight molecules from complex mixtures such as these. In this example, the columns are used for the analysis of alkyl glycerides, and for resolving individual molecules from complex alkyl glyceride mixtures such as occur in frying fats.

Reference

1. P. Wolff, F.X. Mordret, A. Dieffenbacher. *Pure and App. Chem.*, **63**, 1163 (1991).

The samples were made up in tetrahydrofuran and injected without further treatment. Figure 25 shows two overlaid chromatograms of lauryl and stearyl mono-, di-, and triglycerides, illustrating the baseline resolution possible with these high efficiency columns.

Figure 26 shows the separation of a complex mixture of alkyl glycerides. Although baseline resolution was not possible with two columns, the two-column set has resolved the individual components from the mixture, allowing identification of the molecules to be made after appropriate calibration.

Conditions

Columns: 2 x Agilent PLgel 3 μm 100 \AA , 7.5 x 300 mm (p/n PL1110-6320)
 Sample: Alkyl glycerides
 Sample concentration: 0.2% (w/v)
 Eluent: THF
 Injection volume: 20 μL
 Flow rate: 1.0 mL/min
 Instrument: Agilent PL-GPC 50 Integrated GPC/SEC System with DRI

Peak ID

1. Stearyl mono
2. Diglyceride
3. Triglyceride
4. Lauryl mono
5. Diglyceride
6. Triglyceride

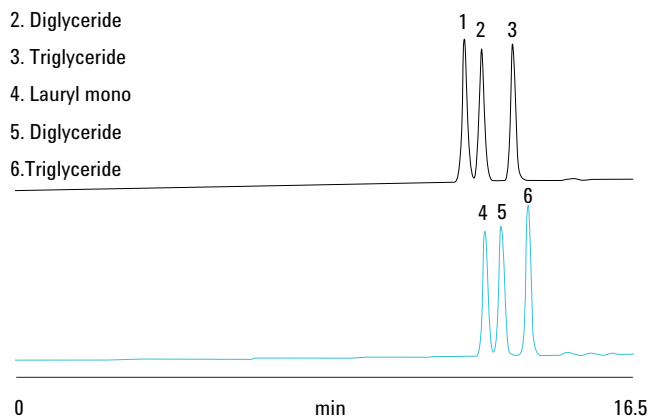


Figure 25. Overlaid chromatograms of lauryl and stearyl mono-, di-, and triglycerides, showing the base line resolution possible with high efficiency Agilent PLgel 3 μm columns.

Peak ID

- | | |
|---|--|
| 1. Tristearoylglyceride
(891.5 g/mol) | 5. Dimyristoylglyceride
(512.8 g/mol) |
| 2. Tripalmitoylglyceride
(807.3 g/mol) | 6. Dilauroylglyceride
(456.7 g/mol) |
| 3. Distearoylglyceride
(635.0 g/mol) | 7. Monopalmitoylglyceride
(330.5 g/mol) |
| 4. Dipalmitoylglyceride
(568.9 g/mol) | 8. Monolauroylglyceride
(274.4 g/mol) |

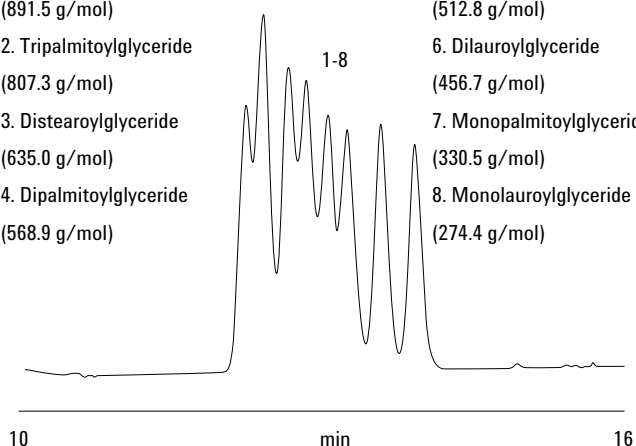


Figure 26. An Agilent PLgel 3 μm two-column set resolves individual components from an alkyl glyceride mixture.

Further resolution could be obtained by adding additional columns, according to the equation:

$$R_{sp} = \frac{0.25}{\sigma D}$$

where R_{sp} is the specific resolution, σ is the peak variance (related to the peak width) and D is the slope of the calibration curve. Increasing the number of columns in the analysis reduces the slope of the calibration curve.



Pesticides

Rapeseed oil (canola oil)

Rapeseed oil (canola oil) is a healthy salad and domestic cooking oil with typically just 7% saturated fat content. The oil is also high in linolenic acid, one of the 'Omega 3' fatty acids identified as having extensive health benefits.

Harvested rapeseed oil can commonly contain traces of pesticides that were used to treat the growing crops. For food safety purposes, pesticide levels must be closely monitored to remove the danger of any adverse health effects. GPC can isolate toxic pesticides from contaminated rapeseed oil. By scaling up these analytical separations to preparative GPC, it is possible to isolate practical quantities of individual components that can be used in further analysis or compound elucidation.

Initially, the optimum loading of the rapeseed oil sample on the columns is analyzed on an analytical scale. The prep scale conditions are determined from the results obtained on the analytical scale.

Analytical chromatograms of rapeseed oil at different concentrations are shown in Figure 27. The chromatograms indicate that the oil and additive could be analyzed at a concentration of 7.0% without serious loss of resolution. The preparative scale separation is shown in Figure 28, at the same oil concentration.



Conditions (analytical scale)

Columns: Agilent PLgel 10 μm 100Å, 7.5 x 300 mm (p/n PL1110-6120)
Sample: Rapeseed oil
Sample concentration: 7.0% (w/v)
Eluent: Dichloromethane
Injection volume: 200 μL
Flow rate: 1.0 mL/min, 1.0 mL/min to the detector
Instrument: Agilent 1260 Infinity Multi-Detector GPC/SEC System, isocratic pump, manual injector, with DRI

Conditions (preparative scale)

Columns: Agilent EnviroPrep, 25 x 300 mm (p/n PL1210-6120EPA)
Sample: Rapeseed oil
Sample concentration: 7.0% (w/v)
Eluent: Dichloromethane
Injection volume: 2 mL
Flow rate: 10.0 mL/min, 0.5 mL/min to the detector
Instrument: Agilent 1260 Infinity Multi-Detector GPC/SEC System, isocratic pump, manual injector, with DRI

Gel permeation chromatography using refractive index detection and EnviroPrep columns is a simple system for the high resolution separation of pesticides in food samples. In this instance, a simple loading study established the prep-scale conditions for a successful isolation of a pesticide in a sample of rapeseed oil.

EnviroPrep columns are packed with high resolution macroporous material ready for use on any preparative high pressure liquid chromatography system. Macroporous materials are highly crosslinked with a rigid and permanent pore structure that ensures high resolution separations, particularly where high sample loading is not required.

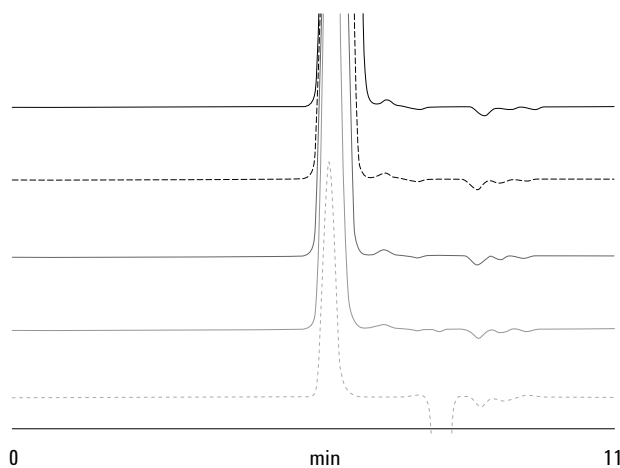


Figure 27. A series of analytical chromatograms of rapeseed oil at concentrations ranging between 1.0 to 7.0% (w/v). The chromatograms show that the oil and additive could be analyzed at a concentration of 7.0% (w/v) without serious loss of resolution.

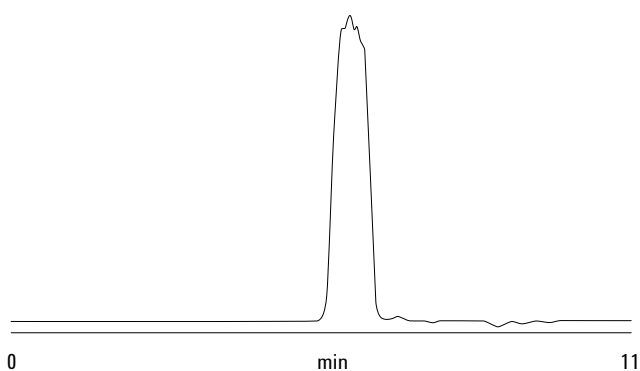


Figure 28. A preparative GPC-scale chromatogram of rapeseed oil at a concentration of 7.0% (w/v).

Mackerel

As part of a food safety program, it was necessary to assess the level of the pesticide hexachlorocyclohexane (1,2,3,4,5,6- $C_6H_6Cl_6$) in a sample of seafood. Gel permeation chromatography was used because GPC is a simple technique for cleaning up food and environmental samples. EnviroPrep columns were selected for the separation. The low pore size of EnviroPrep columns excludes high molecular material, maximizing separation of these interferences from the low molecular weight species of interest. Figure 29 clearly shows that EnviroPrep columns can be used to separate high molecular weight lipids that are excluded on the columns from a small-molecule pesticide. This allowed collection of the pure pesticide in solvent for further analysis.

Conditions

Columns: 2 x Agilent EnviroPrep, 25 x 300 mm (p/n PL1210-6120EPA)
Sample prep: 1 g mackerel macerated and extracted in 50 mL chloroform
Sample: Mackerel
Eluent: Tetrahydrofuran
Flow rate: 10 mL/min
Instrument: Agilent 1260 Infinity Isocratic Pump, manual injector, with DRI

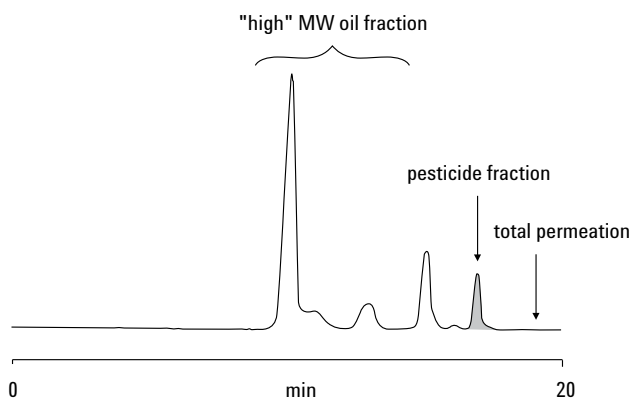


Figure 29. The chromatogram clearly shows that Agilent EnviroPrep columns be used to separate high molecular weight lipids that are excluded on the columns from a small molecule pesticide. This allowed collection of the pure pesticide in solvent for further analysis.

Gel permeation chromatography with EnviroPrep columns is a straightforward system for high resolution separation of pesticides in food samples. In this example, interfering high molecular weight lipids were separated from a low molecular weight pesticide in a sample of mackerel.

To allow the separation of trace components at low concentrations, the rigid and well-defined pores of the EnviroPrep macroporous packing material give unrivalled resolution and peak shape. In addition, for maximum flexibility, the columns can be used with any liquid chromatography system capable of isocratic flow at the required flow rates.





Packaging

In the food industry, one of the largest uses of polymers is in packaging. Polyolefins such as polyethylene are used to make films and molded containers, and properties such as mechanical strength and toughness are vital to the correct performance of such containers in use. GPC is a well-known technique for investigating the properties of polyolefins, and the following applications illustrate this analysis.



High molecular weight polyolefins

Polyolefins range from low molecular weight hydrocarbon waxes to ultra high molecular weight rigid plastics. The molecular weight distributions of polyolefins is directly related to physical properties such as toughness, melt viscosity and crystallinity. High molecular weight polyolefins tend to exhibit very broad molecular weight distribution (MWD). For such samples, small particles with small pore sizes are not desirable since shear degradation may occur, and so the high-pore-size particles of PLgel Olexis are recommended

Conditions

Columns: 3 x Agilent PLgel Olexis, 7.5 x 300 mm (p/n PL1110-6400)
Sample: Polyethylenes
Eluent: TCB + 0.015% BHT
Injection volume: 200 μ L
Flow rate: 1 mL/min
Temperature: 160 $^{\circ}$ C
Instrument: Agilent PL-GPC 220 High Temperature GPC/SEC System with DRI, viscometer

Artifacts known as dislocations can arise in blended columns, resulting from a mismatch of the pore volume of components in the blend. Dislocations lead to false modalities and polydispersities. Avoiding dislocations was an integral part of the design brief for PLgel Olexis columns. Accurate blending of these components produces a column that gives a smooth molecular weight distribution, providing a true reflection of the shape of the MWD (Figure 30). PLgel Olexis is perfect for studies that require accurate polydispersity index and modality information.

Figure 31 shows a range of polyolefin samples analyzed on a PLgel Olexis column, covering the spread of molecular weights. There are no dislocations and the peak shape of the very broad samples shows true sample modality.

Given the accurate resolving power of PLgel Olexis, analysts can be confident that unusual peak shapes are real and not artifacts; unusual peak shapes of some samples will be true reflections of their modality. This is important for studies into reaction mechanisms and catalyst behavior (Figure 32).

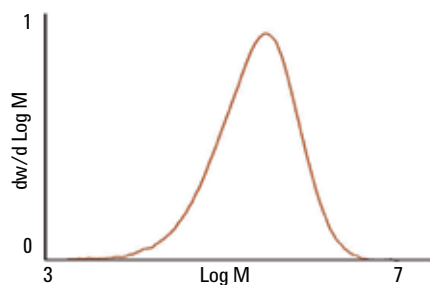


Figure 30. True representation of polyolefin molecular weight distribution with Agilent PLgel Olexis.

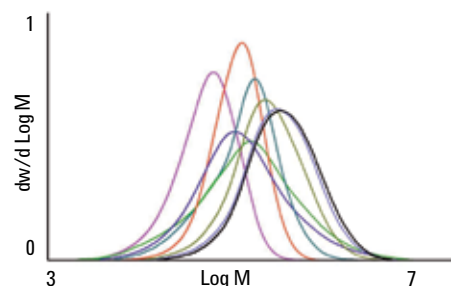


Figure 31. Agilent PLgel Olexis reveals true modalities across the range of polyolefins.

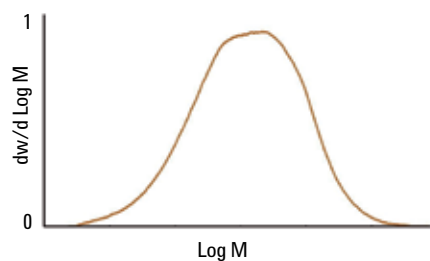


Figure 32. A true change in peak shape revealed by Agilent PLgel Olexis of a multimodal material manufactured from a multisite catalyst.

Lower molecular weight polyolefins

Crude oil, or petroleum, is the main source of organic chemicals for industry. The major chemicals are derived from two constituents of oil, xylene and naphtha. These raw materials are then broken down into more basic products, e.g. polyethylene, polypropylene, elastomers, asphalts, and liquid hydrocarbons.

Characterization of such products is commonly achieved using GPC. However, the diversity of petroleum products demands a variety of GPC column types for optimized analysis. Low molecular weight liquid hydrocarbons require high resolution of individual components. This is illustrated in Figure 33, where three linear hydrocarbons were resolved easily to baseline in a reasonably short analysis time on PLgel 5 μm columns.

Conditions

Columns: 2 x Agilent PLgel 5 μm 100Å, 7.5 x 300 mm (p/n PL1110-6520)
Sample: Linear hydrocarbons
Eluent: TCB
Flow rate: 1 mL/min
Temperature: 145 °C
Instrument: Agilent PL-GPC 220 High Temperature GPC/SEC System

Peak Identification

1. $\text{C}_{36}\text{H}_{64}$
2. $\text{C}_{22}\text{H}_{46}$
3. $\text{C}_{14}\text{H}_{30}$

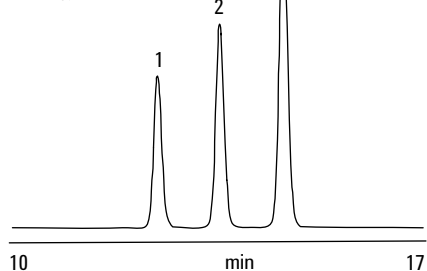


Figure 33. Linear hydrocarbons separated to baseline on an Agilent PLgel column set.

Figure 34 shows the separation of a selection of low molecular weight linear hydrocarbons on PLgel 3 μm columns.

Conditions

Columns: 2 x Agilent PLgel 3 μm 100Å, 7.5 x 300 mm (p/n PL1110-6320)
Sample: Linear hydrocarbons
Eluent: TCB
Injection volume: 20 μL
Flow rate: 0.8 mL/min
Temperature: 145 °C
Instrument: Agilent PL-GPC 220 High Temperature GPC/SEC System

Peak Identification

1. C_{36}
2. C_{24}
3. C_{20}
4. C_{16}
5. C_{12}

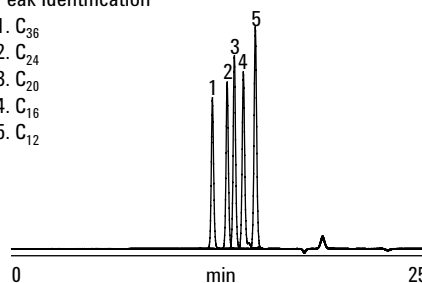


Figure 34. Separation of low molecular weight hydrocarbons.

PLgel 100Å columns have a GPC exclusion limit of 4,000 molecular weight (polystyrene equivalent). Intermediate products can be analyzed using the PLgel MIXED-D column that has a linear molecular weight resolving range up to an exclusion limit of around 400,000 molecular weight. The 5 µm particle size maintains high column efficiency and thus fewer columns are required and analysis time is relatively short. Figure 35 shows a chromatogram of a relatively low molecular weight hydrocarbon obtained on PLgel 5 µm MIXED-D columns.

Conditions

Columns: 2 x Agilent PLgel 5 µm MIXED-D, 7.5 x 300 mm (p/n PL1110-6504)
 Sample: Linear hydrocarbons
 Eluent: TCB
 Injection volume: 200 µL
 Flow rate: 1 mL/min
 Temperature: 160 °C
 Instrument: Agilent PL-GPC 220 High Temperature GPC/SEC System

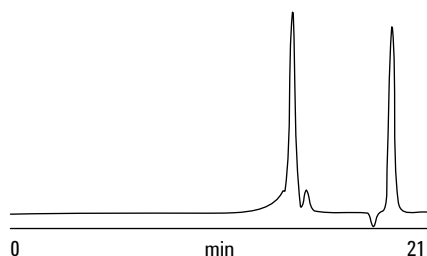


Figure 35. A low molecular weight wax.

High-density polyethylene

High-density polyethylene, or HDPE, has a high strength-to-density ratio, and so it's widely used for making plastic bottles and bottle crates. Analysis of HDPE involves sample preparation at high temperature over an extended period. In this example, a commercial HDPE was prepared at 2 mg/mL using the Agilent PL-SP 260VS Sample Preparation System, with a dissolution temperature of 160 °C and a dissolution time of two hours. Eight aliquots of the master batch solution were dispensed into autosampler vials and placed in the autosampler carousel of the Agilent PL-GPC 220 High Temperature GPC/SEC System. The hot zone temperature was 160 °C and the warm zone 80 °C (Figure 36).

Conditions

Columns: 3 x Agilent PLgel 10 µm MIXED-B, 7.5 x 300 mm (p/n PL1110-6100)
 Eluent: TCB + 0.0125% BHT
 Injection volume: 200 µL
 Flow rate: 1 mL/min
 Temperature: 160 °C
 Instrument: Agilent PL-GPC 220 High Temperature GPC/SEC System

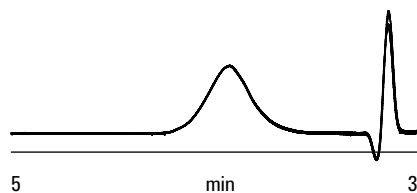


Figure 36. Overlay of the raw data chromatograms obtained for eight consecutive injections of HDPE.

The data were analyzed against a polystyrene standards calibration using the following Mark-Houwink parameters to obtain the polypropylene equivalent molecular weight averages that are shown in Table 2.

¹Polystyrene in TCB $K = 12.1 \times 10^{-5}$ $a = 0.707$

²Polyethylene in TCB $K = 40.6 \times 10^{-5}$ $a = 0.725$

Table 2. Summary of results from eight injections of high-density polyethylene.

Injection number	Mn	Mp	Mw
1	17,289	76,818	333,851
2	16,988	77,434	335,496
3	17,428	77,514	332,616
4	17,521	77,052	335,635
5	17,348	76,520	334,212
6	17,487	77,728	333,511
7	16,898	77,578	335,642
8	17,717	77,288	334,923
Mean	17,302	77,241	334,485
Std Dev	220	387	1,048
% Variation	1.3	0.5	0.3

Figure 37 shows an overlay of the molecular weight distribution calculated for the eight consecutive injections of the HDPE sample, and illustrates the excellent repeatability obtained with the Agilent PL-GPC 220 High Temperature GPC/SEC System using PLgel 10 μ m MIXED-B columns.

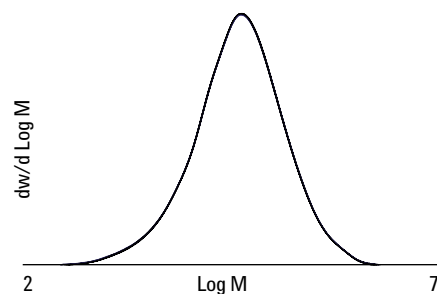


Figure 37. Molecular weight overlay of eight consecutive injections of HDPE.



High-density polypropylene

A similar study was also done on a sample of high-density polypropylene, prepared at 1.5 mg/mL. Other sample preparation was as for HDPE. Figure 38 shows an overlay of the raw data chromatograms obtained for six consecutive injections of the sample.

Conditions

Columns: 3 x Agilent PLgel 10 μ m MIXED-B, 7.5 x 300 mm
(p/n PL1110-6100)
Eluent: TCB + 0.0125% BHT
Injection volume: 200 μ L
Flow rate: 1 mL/min
Temperature: 160 $^{\circ}$ C
Instrument: Agilent PL-GPC 220 High Temperature GPC/SEC System

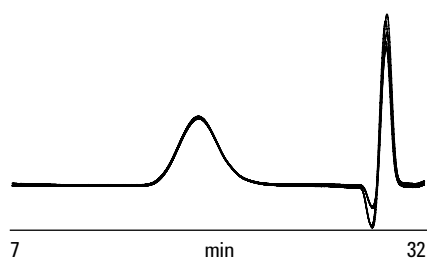


Figure 38. Overlay of the raw data chromatograms obtained for six consecutive injections of HDPP.

Again, data were analyzed against a polystyrene standards calibration using the following Mark-Houwink parameters to obtain the polypropylene-equivalent molecular weight averages shown in Table 3.

¹Polystyrene in TCB $K = 12.1 \times 10^{-5}$ $\alpha = 0.707$

²Polypropylene in TCB $K = 19.0 \times 10^{-5}$ $\alpha = 0.725$

Table 3. Overlay of the raw data chromatograms obtained for six consecutive injections of high-density polypropylene.

Injection number	M _n	M _p	M _w
1	127,132	65,086	185,795
2	131,893	65,089	185,236
3	128,673	66,802	186,202
4	132,062	67,417	188,048
5	131,625	69,320	188,679
6	130,227	69,677	186,188
Mean	130,202	67,232	186,691
Std Dev	1,693	1,815	1,239
% Variation	0.13	2.70	0.66

Figure 39 shows an overlay of the molecular weight distribution calculated for the six consecutive injections of the HDPP sample, with excellent repeatability.

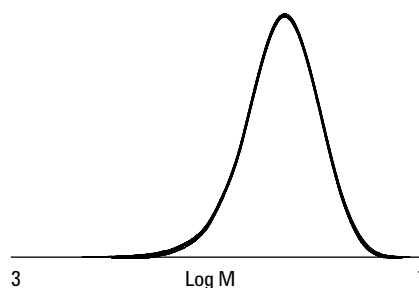


Figure 39. Molecular weight overlay of six consecutive injections of HDPP.

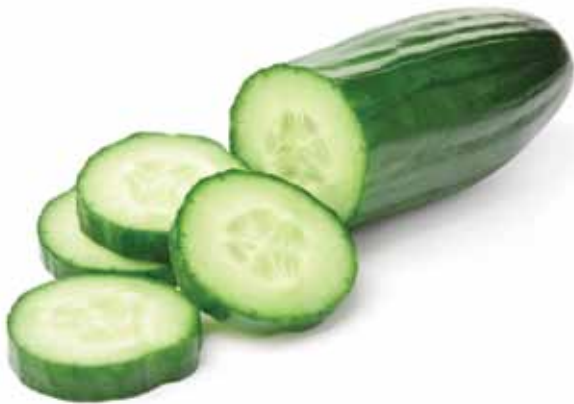
References

1. H. Coll, D. K. Gilding. *J. Poly. Sci. Pt A-2: Poly. Phys.* **8**, 89 (1970).
2. T. G. Scholte, N. L. J. Meijerink, H. M. Schoffeleers, A.M.G. Brands. *J. Appl. Poly. Sci.* **29**, 3763 (1984).



Wax coatings

The term wax is used to describe a wide range of materials that share a similar appearance and consistency. Typically, waxes are white or tan in color and range from soft, readily pliable materials to harder, more resistant products.



Waxes are generally of two types; natural, renewable waxes such as carnauba or beeswax, and crude oil products such as paraffin waxes. Both types are used in the food industry as protective coatings on fruit, vegetables, or candy. Beeswax, for example, is used as an edible coating for cheese, to protect the food as it ages.

The solubility of waxes is very dependent on molecular weight. Lower molecular weight waxes are soluble in tetrahydrofuran. However, as the molecular weight increases, the wax becomes harder and more brittle (due to higher crystallinity) and more aggressive solvents such as trichlorobenzene may be required for dissolution. THF is an appropriate eluent for the GPC of beeswax.

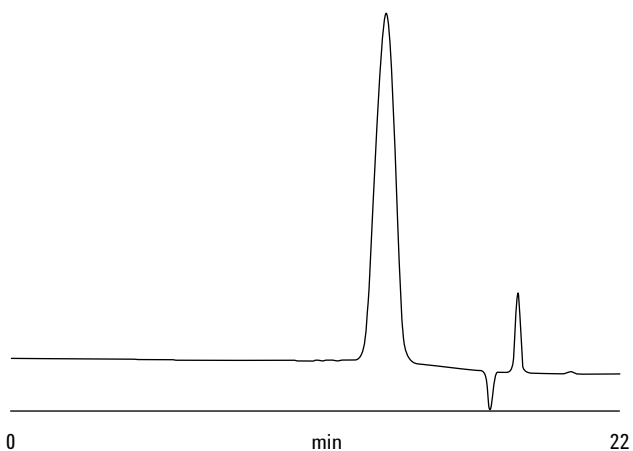


Figure 40. Chromatogram of beeswax on an Agilent PLgel 3 μm two-column set.

Beeswax is a complex product made from the honeycomb of bees. The wax is firm and yellow in color and has been known for thousands of years, with a wide range of applications. Beeswax typically contains about 15% partially unsaturated hydrocarbons, 15% free fatty acids, and 70% monohydroxyesters and di- and tripolyesters.

Conditions

Columns: 2 x Agilent PLgel 3 μm 100 \AA , 7.5 x 300 mm (p/n PL1110-6320)
 Sample: Beeswax
 Eluent: THF (stabilized)
 Injection volume: 20 μL
 Flow rate: 1.0 mL/min
 Instrument: Agilent 1260 Infinity Multi-Detector GPC/SEC System, isocratic pump, manual injector, with DRI

Beeswax elutes as a broad polymer peak indicating that the various components had a similar size in solution (Figure 40).

GPC/SEC System Configurations

The following Tables (4 to 13) will assist you in selecting the right system for your application. They show which components are required, and which are optional.

Agilent 1260 Infinity GPC/SEC System

Table 4. Sample delivery module requirements by application, for the Agilent 1260 Infinity GPC/SEC system.



		Sample Delivery Modules			
		G1310B	G1322A	G1316A	G1329B
		1260 Infinity Isocratic Pump	1260 Infinity Standard Degasser	1260 Infinity Thermostatted Column Compartment	1260 Infinity Standard Autosampler or
					G1328C
					1260 Infinity Manual Injector
Application		GPC/SEC only requires isocratic flow	Solvent degassing recommended for GPC	Up to 80 °C TCC accomodates two 7.5 x 300 mm GPC columns	Typical injection volumes in GPC/SEC are 20, 50, 100 and 200 µL
Additives, contaminants	eg. Triglycerides, pesticides	✓	✓	✓	✓
Natural and synthetic rubbers	eg. Gum arabic, polybutadiene, polysiloxane, polybutadiene, polyisoprene, SBR, silicone	✓	✓	✓	✓
Polyesters and Polyamides	eg. Polyethylene terephthalate, polylactic acid, polyamide, nylon	✓	✓	✓	✓
Polysaccharides	eg. Cellulose derivatives, chitin, pectin, starches, pullulan, dextran	✓	✓	✓	✓
Resins	eg. Epoxies, polyurethane, acrylate polymers, phenolics	✓	✓	✓	✓
Vinyl polymers	eg. Polystyrene, polyvinyl chloride, polyvinyl acetate, polyvinyl alcohol, Polyacrylonitrile	✓	✓	✓	✓
Waxes	eg. Beeswax, carnauba, paraffin wax	✓	✓	✓	✓
UV Absorbing Polymers	eg. Polystyrene, poly(styreneacrylonitrile) polymethylmethacrylate polybutadienes, polycarbonates, polyacrylic acids	✓	✓	✓	✓

Key

✓ Required

✓ Optional

Table 5. Detector and software requirements by application, for the Agilent 1260 Infinity GPC/SEC system.



Application		Detectors		Control, Collection & Analysis Software	
		G1362A 1260 Infinity Refractive Index Detector Includes 8 µL flow cell and LAN interface	G1314F 1260 Infinity Variable Wavelength Detector or G1365D 1260 Infinity Multiple Wavelength Detector For single or multi-wavelength analysis, only one channel collected	G7850AA Agilent GPC/SEC Software Standalone software dedicated to GPC calculations. Analysis only.	G7854AA Agilent GPC/SEC Instrument Drivers Provides Instrument Control and Data Collection.
Additives, contaminants	eg. Triglycerides, pesticides	✓		✓	✓
Natural and synthetic rubbers	eg. Gum arabic, polybutadiene, polysiloxane, polybutadiene, polyisoprene, SBR, silicone	✓		✓	✓
Polyesters and Polyamides	eg. Polyethylene terephthalate, polylactic acid, polyamide, nylon	✓		✓	✓
Polysaccharides	eg. Cellulose derivatives, chitin, pectin, starches, pullulan, dextran	✓		✓	✓
Resins	eg. Epoxies, polyurethane, acrylate polymers, phenolics	✓		✓	✓
Vinyl polymers	eg. Polystyrene, polyvinyl chloride, polyvinyl acetate, polyvinyl alcohol, polyacrylonitrile	✓		✓	✓
Waxes	eg. Beeswax, carnauba, paraffin wax	✓		✓	✓
UV Absorbing Polymers	eg. Polystyrene, poly(styreneacrylonitrile) polymethylmethacrylate polybutadienes, polycarbonates, polyacrylic acids	✓	✓	✓	✓

Key

✓ Required

✓ Optional

Agilent 1260 Infinity Multi-Detector GPC/SEC System

Table 6. Sample delivery module requirements by application, for the Agilent 1260 Infinity Multi-Detector GPC/SEC System.




Application		Sample Delivery Modules			
		G1310B	G1322A	G1316A	G1329B
		1260 Infinity Isocratic Pump	1260 Infinity Standard Degasser	1260 Infinity Thermostatted Column Compartment	1260 Infinity Standard Autosampler or
					G1328C 1260 Infinity Manual Injector
		GPC/SEC only requires isocratic flow	Solvent degassing recommended for GPC	Up to 80 °C TCC accommodates two 7.5 x 300 mm GPC columns	Typical injection volumes in GPC/SEC are 20, 50, 100 and 200 µL
Additives, contaminants	eg. Triglycerides, pesticides	✓	✓	✓	✓
Natural and synthetic rubbers	eg. Gum arabic, polybutadiene, polysiloxane, polybutadiene, polyisoprene, SBR, silicone	✓	✓	✓	✓
Polyesters and Polyamides	eg. Polyethylene terephthalate, polylactic acid, polyamide, nylon	✓	✓	✓	✓
Polysaccharides	eg. Cellulose derivatives, chitin, pectin, starches, pullulan, dextran	✓	✓	✓	✓
Resins	eg. Epoxies, polyurethane, acrylate polymers, phenolics	✓	✓	✓	✓
Vinyl polymers	eg. Polystyrene, polyvinyl chloride, polyvinyl acetate, polyvinyl alcohol, polyacrylonitrile	✓	✓	✓	✓
Waxes	eg. Beeswax, carnauba, paraffin wax	✓	✓	✓	✓
UV Absorbing Polymers	eg. Polystyrene, poly(styreneacrylonitrile) polymethylmethacrylate polybutadienes, polycarbonates, polyacrylic acids	✓	✓	✓	✓

Key

✓ Required

✓ Optional

Table 7. Detector requirements by application, for the Agilent 1260 Infinity Multi-Detector GPC/SEC System.




		Detectors				
		G7800A	G7800A#031	G7800A#032	G7800A#033	G1314F
		1260 Infinity GPC/SEC Multi-Detector Suite	1260 Infinity GPC/SEC Refractive Index Detector	1260 Infinity GPC/SEC Viscometer	1260 Infinity GPC/SEC Dual Angle Light Scattering Detector	1260 Infinity Variable Wavelength Detector or G1365D 1260 Infinity Multiple Wavelength Detector
		Includes integrated control module for data collection and manual control	Refractive index detector, most common detector for GPC	Viscometer, used to generate the Universal Calibration	Dual angle Light Scattering detector, 15 & 90°, for absolute Mw	For single or multi-wavelength analysis, only one channel collected
Application						
Additives, contaminants	eg. Triglycerides, pesticides	✓	✓			
Natural and synthetic rubbers	eg. Gum arabic, polybutadiene, polysiloxane, polybutadiene, polyisoprene, SBR, silicone	✓	✓	✓	✓	
Polyesters and Polyamides	eg. Polyethylene terephthalate, polylactic acid, polyamide, nylon	✓	✓	✓	✓	
Polysaccharides	eg. Cellulose derivatives, chitin, pectin, starches, pullulan, dextran	✓	✓	✓	✓	
Resins	eg. Epoxies, polyurethane, acrylate polymers, phenolics	✓	✓			
Vinyl polymers	eg. Polystyrene, polyvinyl chloride, polyvinyl acetate, polyvinyl alcohol, polyacrylonitrile	✓	✓	✓	✓	
Waxes	eg. Beeswax, carnauba, paraffin wax	✓	✓			
UV Absorbing Polymers	eg. Polystyrene, poly(styreneacrylonitrile) polymethylmethacrylate polybutadienes, polycarbonates, polyacrylic acids	✓	✓			✓

Key

✓ Required

✓ Optional

Table 8. Software requirements by application, for the Agilent 1260 Infinity Multi-Detector GPC/SEC System.



		Control, Collection & Analysis Software		
		G7850AA	G7852AA	G7854AA
		Agilent GPC/SEC Software	Agilent GPC/SEC Multi-Detector upgrade	Agilent GPC/SEC Instrument Drivers
		Standalone software dedicated to GPC calculations. Analysis only.	Upgrade to Agilent GPC/SEC software dedicated to multi-detector GPC calculations.	Provides Instrument Control and Data Collection.
Application				
Additives, contaminants	eg. Triglycerides, pesticides	✓		✓
Natural and synthetic rubbers	eg. Gum arabic, polybutadiene, polysiloxane, polybutadiene, polyisoprene, SBR, silicone	✓	✓	✓
Polyesters and Polyamides	eg. Polyethylene terephthalate, polylactic acid, polyamide, nylon	✓	✓	✓
Polysaccharides	eg. Cellulose derivatives, chitin, pectin, starches, pullulan, dextran	✓	✓	✓
Resins	eg. Epoxies, polyurethane, acrylate polymers, phenolics	✓		✓
Vinyl polymers	eg. Polystyrene, polyvinyl chloride, polyvinyl acetate, polyvinyl alcohol, polyacrylonitrile	✓	✓	✓
Waxes	eg. Beeswax, carnauba, paraffin wax	✓		✓
UV Absorbing Polymers	eg. Polystyrene, poly(styreneacrylonitrile) polymethylmethacrylate polybutadienes, polycarbonates, polyacrylic acids	✓		✓

Key
 ✓ Required
 ✓ Optional

Agilent PL-GPC 220 High Temperature GPC/SEC System

Table 9. Sample delivery modules, software, sample preparation and detector requirements by application, for the Agilent PL-GPC 220 High Temperature System.

Application		Sample Delivery Modules			Additional Detectors	
		G7820A			G7820A#032	G7820A#032
		PL-GPC 220 Integrated High Temperature GPC/SEC System	Complete basic system includes degasser, pump, autosampler, oven and RI detector.		PL-GPC 220 Viscometer	PL-GPC 220 Dual Angle Light Scattering Detector
				Viscometer, used to generate the Universal Calibration. Housed inside oven.	Dual angle Light Scattering detector, 15 & 90°, for absolute Mw. Housed inside oven.	
Polyamides	eg. Polyamide, nylon	✓		✓		✓
Polyolefins	eg. Polyethylene, polypropylene, Poly(ethylene -co - norbornene)	✓		✓		✓
Waxes	eg. Polyethylene wax	✓		✓		✓
EVA copolymers	eg. Vinyl acetate ethylene copolymer	✓		✓		✓
PBT	eg. Epoxies, polyurethane, acrylate polymers, phenolics	✓		✓		✓
Application		Control, Collection & Analysis Software			Sample Preparation	
		G7850AA	G7852AA	G7820A#040	G7820A#065	
		Agilent GPC/SEC Software	Agilent GPC/SEC Multi-Detector upgrade	PL DataStream	PL-SP 260VS Sample Prep System	
		Standalone software dedicated to GPC calculations. Analysis only.	Upgrade to Agilent GPC/SEC software dedicated to multi-detector GPC calculations.	Required for data collection of up to 4 channels of data (A/D converter).	Sample prep & filtration system, with agitation and heating to 250°C.	
Polyamides	eg. Polyamide, nylon	✓	✓	✓		✓
Polyolefins	eg. Polyethylene, polypropylene, Poly(ethylene -co - norbornene)	✓	✓	✓		✓
Waxes	eg. Polyethylene wax	✓	✓	✓		✓
EVA copolymers	eg. Vinyl acetate ethylene copolymer	✓	✓	✓		✓
PBT	eg. Epoxies, polyurethane, acrylate polymers, phenolics	✓	✓	✓		✓
PPS	Polybutylene, terephthalate	✓	✓	✓		✓


Key

✓ Required

✓ Optional

Agilent PL-GPC 50 Integrated GPC/SEC System

Table 10. Sample delivery module requirements by application, for the Agilent PL-GPC 50 Integrated GPC/SEC System.




		Sample Delivery Modules		
		G7810A	G7810A#011	G7810A#060
		PL-GPC 50 Integrated GPC/SEC System	PL-GPC 50 with degasser	PL-GPC 50 Autosampler
		Complete basic system, including pump, injection valve, oven and RI detector.	With added internal degasser. Cannot be retro-fitted. Highly Recommended.	56 vial positions. Available as 2 mL & 4 mL split tray.
Application				
Additives, contaminants	eg. Triglycerides, pesticides	✓	✓	✓
Natural and synthetic rubbers	eg. Gum arabic, polybutadiene, polysiloxane, polybutadiene, polyisoprene, SBR, silicone	✓	✓	✓
Polyesters and Polyamides	eg. Polyethylene terephthalate, polylactic acid, polyamide, nylon	✓	✓	✓
Polysaccharides	eg. Cellulose derivatives, chitin, pectin, starches, pullulan, dextran	✓	✓	✓
Resins	eg. Epoxies, polyurethane, acrylate polymers, phenolics	✓	✓	✓
Vinyl polymers	eg. Polystyrene, polyvinyl chloride, polyvinyl acetate, polyvinyl alcohol, polyacrylonitrile	✓	✓	✓
Waxes	eg. Beeswax, carnauba, paraffin wax	✓	✓	✓

Key

✓ Required

✓ Optional

Table 11. Detector and software requirements by application, for the Agilent PL-GPC 50 Integrated GPC/SEC System.



		Additional Detectors		Control, Collection & Analysis Software		
		G7810A#032	G7810A#033	G7850AA	G7852AA	G7854AA
		PL-GPC 50 Viscometer	PL-GPC 50 Dual Angle Light Scattering Detector	Agilent GPC/SEC Software	Agilent GPC/SEC Multi-Detector upgrade	Agilent GPC/SEC Instrument Drivers
		Viscometer, used to generate the Universal Calibration. Housed within PL-GPC 50 unit.	Dual angle Light Scattering detector, 15 & 90°, for absolute Mw. Housed within PL-GPC 50 unit.	Standalone software dedicated to GPC calculations. Analysis only.	Upgrade to Agilent GPC/SEC software dedicated to multi-detector GPC calculations	Provides Instrument Control and Data Collection.
Application						
Additives, contaminants	eg. Triglycerides, pesticides			✓		✓
Natural and synthetic rubbers	eg. Gum arabic, polybutadiene, polysiloxane, polybutadiene, polyisoprene, SBR, silicone	✓	✓	✓	✓	✓
Polyesters and Polyamides	eg. Polyethylene terephthalate, polylactic acid, polyamide, nylon	✓	✓	✓	✓	✓
Polysaccharides	eg. Cellulose derivatives, chitin, pectin, starches, pullulan, dextran	✓	✓	✓	✓	✓
Resins	eg. Epoxies, polyurethane, acrylate polymers, phenolics			✓		✓
Vinyl polymers	eg. Polystyrene, polyvinyl chloride, polyvinyl acetate, polyvinyl alcohol, polyacrylonitrile	✓	✓	✓	✓	✓
Waxes	eg. Beeswax, carnauba, paraffin wax			✓		✓

Key

✓ Required

✓ Optional



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