Analysis of engineering polymers by GPC/SEC

Application Compendium

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The Measure of Confidence

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Polymer analysis by GPC/SEC

Introduction

Increasingly, plastics are being utilized to perform structural and mechanical roles in the construction and engineering industries. The favorable properties of polymers, such as mechanical strength, durability, and resistance to chemical and physical degradation, coupled with their relative cheapness, means that polymers outperform many traditional materials, including wood and metals, in key applications. With the creation of new polymeric materials, this shift towards plastics is becoming even more pronounced as materials with new properties are designed and developed. An understanding of the behavior of polymers is key to designing new materials with appropriate performance characteristics for specific applications. Analysis of these materials is therefore a critical component of the development and manufacture of engineering polymers.

Gel permeation chromatography (GPC, also known as size exclusion chromatography, SEC) is a well-known technique for assessing the molecular weight distribution of polymers, a property that influences many of their physical properties. Generally, increasing molecular weight leads to higher performance characteristics, while an increase in the width of the distribution (the polydispersity) leads to a loss of performance but an increase in the ease of processing.

Engineering polymers are particularly difficult to analyze – they are generally tough and difficult to dissolve, often requiring aggressive solvents and elevated temperatures. For these applications at high temperature, a high performance integrated GPC system, such as the Agilent PL-GPC 220 Integrated GPC/SEC System, is a necessity. The PL-GPC 220 has the highest temperature range of any system on the market. The following applications show the analysis of various types of engineering polymers and illustrate the conditions and equipment required.

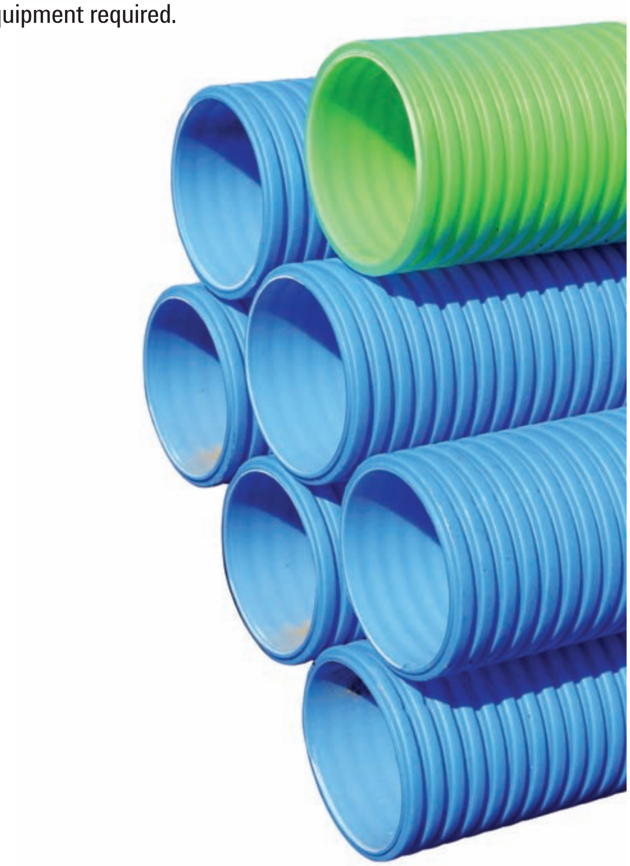

Table 1. *Effects of molecular weight distribution on the properties of engineering polymers*

	Strength	Toughness	Brittleness	Melt viscosity	Chemical resistance	Solubility
Increasing Mw	+	+	+	+	+	–
Decreasing distribution	+	+	–	+	+	+

Agilent's GPC/SEC technology

Agilent produces the most extensive range of GPC columns, standards, instruments and software, ideally suited to the analysis of engineering polymers.

GPC/SEC columns

Agilent's GPC columns are the most rugged and reliable on the market, making them ideal for applications performed in viscous solvents and at elevated temperatures where column lifetime can be an issue. The extensive column range includes products tailored to the analysis of engineering polymers that generally have high molecular weights and high viscosities, and includes specialist columns such as Agilent PLgel Olexis that are designed for the analysis of a specific material. With extensive options in particle and pore size, Agilent's columns can be selected to match the molecular weight of the material under investigation, thereby ensuring that the best quality of data is obtained from the GPC/SEC experiment.

Agilent offers a selection of GPC/SEC column dimensions

GPC/SEC standards

Narrow polydispersity polymer standards with very highly characterized molecular weights are used as calibration standards in the GPC analysis of engineering polymers. Polystyrene standards are the first choice for many organic solvents, either for conventional GPC column calibration or for calibrating light scattering and viscosity detectors.

Agilent's EasiVial calibration kit

GPC/SEC instruments

Complementing Agilent's column technology is the most extensive collection of integrated GPC/SEC instrumentation on the market, covering the widest temperature range available, from ambient to 220 °C.

Agilent's PL-GPC 220 Integrated GPC/SEC System features unbeatable reproducibility for any GPC/SEC application, across the entire operating range. The PL-GPC 220 is an extremely flexible system, designed to run almost all polymer, solvent and temperature combinations, from 30 to 220 ºC.

The PL-GPC 220 allows all forms of the GPC/SEC experiment to be performed and can be used to analyze the complete range of engineering materials, including those that require analysis at extremely high temperatures. 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 engineering polymers to be analyzed in detail. Agilent's complete range of columns and instrumentation offer a clear advantage in the analysis of engineering polymers.

PL-GPC 220 Integrated GPC/SEC System

For more information on Agilent's GPC/SEC products, visit **www.agilent.com/chem/gpcsec**

GPC/SEC of polymers in aggressive solvents

Aggressive solvents

Many polymers, especially those used in engineering applications, show only limited solubility in a small number of solvents. This is because high strength and toughness are usually a result of high molecular weight and/or high crystallinity. Increasing molecular weight requires untangling the molecular chains to dissolve the material, whereas increased crystallinity requires break-up of any inter-chain bonds that may be present.

The PL-GPC 220 Integrated GPC/SEC System is designed to allow the use of even the most aggressive solvents. The following applications illustrate the analysis of a range of engineering polymers that require aggressive solvents for solubility or as eluents during the analysis.

GPC analysis of polyether ether ketone (PEEK)

Application areas: High performance components, tubing in liquid chromatography

Polyether ether ketone (PEEK) was developed in 1977 by ICI and was one of the first of the new generation of engineering thermoplastics developed for chemical resistance, high mechanical strength and high thermal stability – the useful properties of the material are retained up to temperatures as high as 315 °C. A crystalline material with repeat units of two ethers and a ketone group in the polymer backbone, PEEK is a high cost material. For many applications, such as the manufacture of piston components in engines, the insulation of cables and the production of high performance aircraft parts, this cost is justified as there are no other plastics that can offer the same performance properties. The industrial performance of PEEK makes analysis of this material by GPC difficult. PEEK has excellent chemical resistance and is unaffected by many organic and inorganic chemicals, dissolving only in strong or concentrated anhydrous oxidizing agents. Previous methods for analyzing PEEK have involved mixtures of trichlorobenzene and phenol running at high temperatures.

For this analysis, the PEEK sample was dissolved in a small volume of dichloroacetic acid at 120 °C for two hours. After dissolution, the sample was diluted to the required concentration of 0.2% (w/v) with chloroform and injected into a system running at temperature after filtration to remove undissolved material.

The PEEK sample eluted as a broad polymer peak with an MW of 70,000 g/mol and a polydispersity of 2.2. The large system peak observed at the end of the run was due to the excess dichloroacetic acid used in the preparation of the sample.

Conditions

Sample:	Polyether ether ketone (PEEK)
Columns:	2 x Agilent PLgel 10 µm MIXED-B, 300 x 7.5 mm (Part No. PL1110-6100)
Eluent:	80% chloroform 20% dichloroacetic acid
Flow Rate:	1.0 mL/min
Inj Vol:	200 µL
Detector:	PL-GPC 220

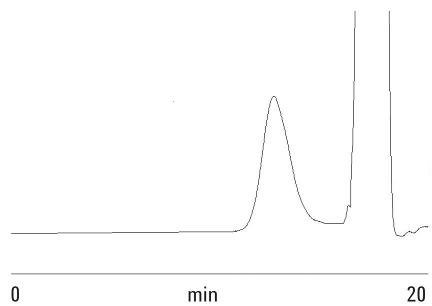

Figure 1. *Chromatogram of a PEEK sample*

GPC analysis of polybutylene terephthalate (PBT) resins in HFIP

Application areas: Machined parts

Polybutylene terephthalate (PBT) resins are used in a wide variety of applications in which toughness and resistance to damage are highly advantageous. However, mechanical and thermal stress during the production of molded parts can cause degradation, giving a reduction in desirable physical properties. The molecular weight distribution of the resin is a key measure of the onset of degradation and therefore of estimating the mechanical strength of the final product. PBT is soluble in 1,1,1,3,3,3-hexafluoroisopropanol (HFIP), a polar organic solvent, which is excellent for dissolving polar polymers such as polyamides and polyesters. The analysis was carried out in HFIP modified by the addition of 20 mM sodium trifluoroacetate to prevent aggregation. Two Agilent PL HFIPgel columns, designed specifically for HFIP applications, were employed for the analysis at a temperature of 40 °C. The PL-GPC 220 Integrated GPC/SEC System was used with differential refractive index and viscometry detection. GPC coupled with a molecular weight-sensitive viscometer allowed calculation of molecular weights based on hydrodynamic volume using the Universal Calibration approach, leading to molecular weights independent of the standards used to generate the column calibration. Agilent polymethylmethacrylate (PMMA) standards were employed to generate the Universal Calibration.

Table 2 shows the molecular weight averages and intrinsic viscosity for the sample before and after molding, as determined by GPC/viscometry. Clearly, the molecular weight distribution indicates that after molding, the material has suffered from degradation and is less robust than the virgin material.

Table 2. *Molecular weight averages and intrinsic viscosity for the PBT resin sample*

	Mn/g mol^{-1}	Mw/g mol^{-1}	Intrinsic viscosity/g^{-1}
Virgin resin	24,400	48,600	0.535
Molded part	11,200	24,000	0.306

Conditions

Samples: PBT resin
Columns: 2 x PL HFIPgel, 300 x 7.5 mm (Part No. PL1114-6900HFIP)
Eluent: HFIP + 20 mM NaTFA
Flow Rate: 1.0 mL/min
Inj Vol: 200 µL
Temp: 40 °C
Detectors: PL-GPC 220, viscometer

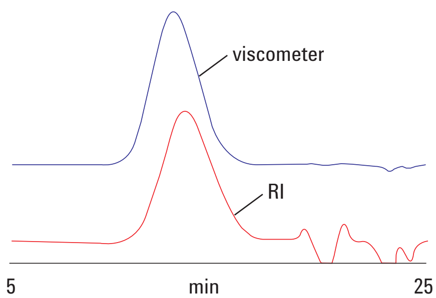

Figure 2. *Example overlay of a dual-detector chromatogram of the virgin PBT resin before molding*

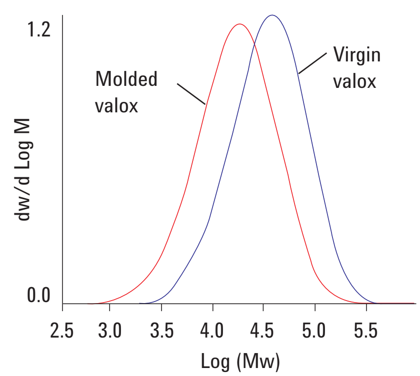

Figure 3. *Molecular weight distributions of the two samples*

Polyethylene terephthalate analysis in o-chlorophenol as an alternative solvent

As an alternative to the use of HFIP, PET can be analyzed in o-chlorophenol. This viscous solvent requires elevated temperatures and is a hazardous substance.

The samples were dissolved by heating to 110 °C for 30 minutes. The polymer remains in solution at room temperature but the high viscosity of the eluent means that high temperature GPC is necessary. Three grades of PET, with different intrinsic viscosities, were analyzed and compared, showing minor differences between the materials.

Conditions

Samples: PET resin
Columns: 2 x PLgel 10 µm MIXED-B, 300 x 7.5 mm (Part No. PL1110-6100)
Eluent: o-chlorophenol
Flow Rate: 1.0 mL/min
Temp: 100 °C
Detection: PL-GPC 220

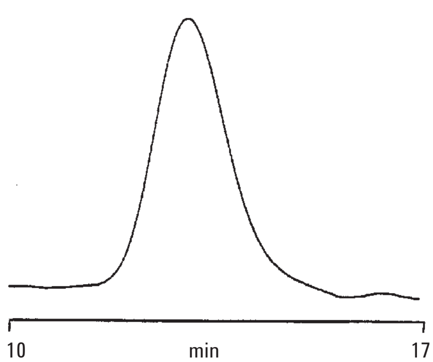

Figure 4. *Chromatogram of a PET sample*

Peak Identification
1. IV=0.72
2. IV=0.75
3. IV=0.84

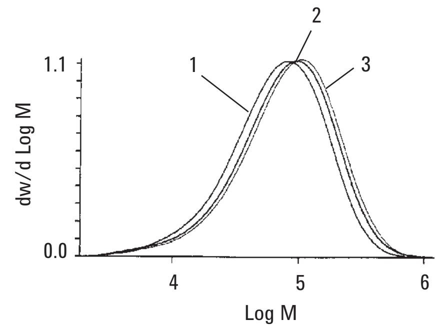

Figure 5. *Molecular weight distributions of the PET samples*

GPC/SEC of polymers at high temperature

High temperature analysis

Some highly crystalline polymers such as polyethylene show only limited solubility and only then at high temperatures. This is due to the fact that temperature is required to break down the ordered crystalline structure, and on cooling, the material will re-crystallize and precipitate from solution. For these applications, high temperature is required throughout the entire analysis to ensure that the samples remain in solution during the experiment. The PL-GPC 220 Integrated GPC/SEC System is capable of maintaining a constant temperature up to 200 °C between the point of injection, the columns and the detector cells, until the point of elution. The following applications illustrate the analysis of crystalline polymers at high temperatures using the PL-GPC 220.

Column selection for polyolefin analysis

Polyolefins range from low molecular weight hydrocarbon waxes to ultra high molecular weight rigid plastics. The molecular weight distribution of polyolefins is directly related to physical properties such as toughness, melt viscosity, and crystallinity. GPC/SEC is widely accepted as the preferred technique to fully characterize the molecular weight distribution of polyolefins.

The selection of a column set for the analysis of a polyolefin is dependent on the molecular weight range of the sample. Low molecular weight samples can be analyzed using high efficiency, relatively low pore size columns. Higher molecular weight materials require large particle size media to minimize shear effects, with a wide pore size distribution.

Figures 6 to 8 show typical data for four very different polyolefin samples, all obtained with the PL-GPC 220.

Conditions

Sample:	Linear hydrocarbons
Columns:	2 x Agilent PLgel 3 µm 100Å, 300 x 7.5 mm (Part No. PL1110-6320)
Eluent:	TCB
Flow Rate:	0.8 mL/min
Inj Vol:	20 µL
Temp:	145 °C
Detector:	PL-GPC 220

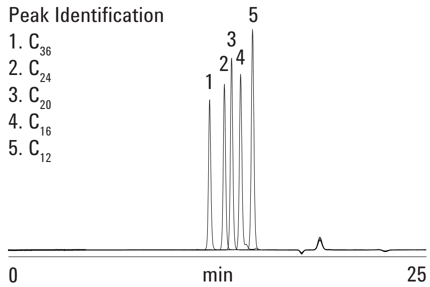

Figure 6. *Separation of a selection of low molecular weight linear hydrocarbons analyzed using two PLgel 3 µm 100Å columns*

Conditions

Sample:	Hydrocarbon wax
Columns:	2 x Agilent PLgel 5 µm MIXED-D, 300 x 7.5 mm (Part No. PL1110-6504)
Eluent:	TCB
Flow Rate:	1.0 mL/min
Inj Vol:	100 µL
Temp:	160 °C
Detector:	PL-GPC 220

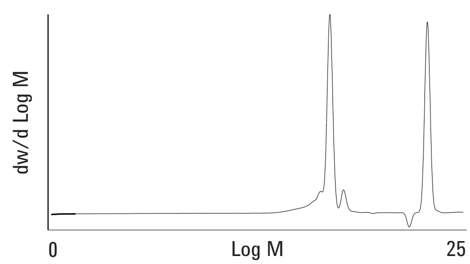

Figure 7. *Chromatogram of a relatively low molecular weight hydrocarbon wax obtained on two PLgel 5 µm MIXED-D columns*

Conditions

Sample: Polyethylene
Columns: 3 x PLgel Olexis, 300 x 7.5 mm (Part No. PL1110-6400)
Eluent: TCB
Flow Rate: 1.0 mL/min
Inj Vol: 200 µL
Temp: 160 °C
Detector: PL-GPC 220

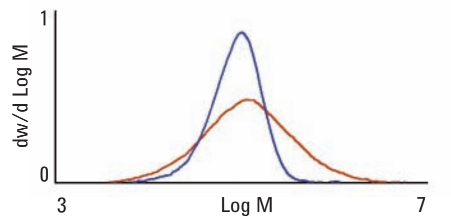

Figure 8. *Overlaid molecular weight distributions of medium and high polydispersity polyethylene samples*

High molecular weight materials require analysis on high pore size PLgel Olexis columns that minimize shear degradation, and are able to resolve up to 100,000,000 g/mol with a 13 µm particle size. These applications illustrate the diversity of polyolefin samples and indicate the flexibility of the PLgel series of columns in addressing the analysis of such samples.

High temperature GPC analysis of polypropylene on the PL-GPC 220 – repeatability study

Application areas: Plastic pipes, bottles and containers

The PL-GPC 220 Integrated GPC/SEC System is ideally suited to the analysis of polypropylene. A commercial sample of polypropylene (PP) was prepared at 1.5 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. Six aliquots of the master batch solution were dispensed into the PL-GPC 220 autosampler vials and placed in the carousel where the hot zone temperature was 160 °C and the warm zone 80 °C.

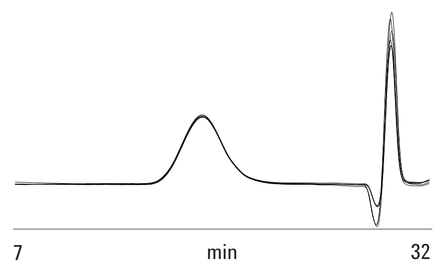

Figure 9. *Overlay of the raw data chromatograms obtained for six consecutive injections of polypropylene*

The data was 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 3.

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

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

Table 3. *Calculated molecular weights for six injections of polypropylene and calculated % variation*

Injection Number	Mp	Mn	Mw
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

The results illustrate the excellent repeatability obtained with the PL-GPC 220 using PLgel 10 µm MIXED-B columns.

Conditions

Sample: Polypropylene
Columns: 3 x PLgel Olexis, 300 x 7.5 mm (Part No.PL1110-6400)
Eluent: TCB + 0.0125% BHT
Flow Rate: 1.0 mL/min
Inj Vol: 200 µL
Temp: 160 °C
Detector: PL-GPC 220

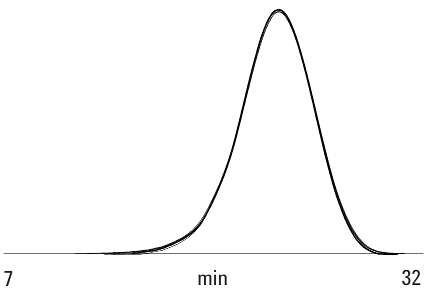

Figure 10. *Overlay of the molecular weight distribution calculated for six consecutive injections of the polypropylene sample*

References

[1] H. Col and D. K. Giddings, *J. Polym. Sci.*, (A2) **8** (1970) 89

[2] T.G. Scholte et al, *J. Appl. Polym. Sci.*, **29** (1984) 3763

Branching analysis of polyethylenes with Cirrus GPC Multi Detector Software

Application areas: Plastic bags and containers

The presence of long chain branching (over 6 carbons in length) in polyolefins strongly influences physical properties such as melt viscosity and mechanical strength. The distribution chain branches in polyolefins are determined by the polymerization mechanism and there is significant interest in the production of materials with well-defined and characterized molecular weight and branching distributions for specific applications.

Here we describe the analysis of three samples of polyethylene with the PL-GPC 220 by GPC/viscometry. Two of the samples had been synthesized by a mechanism to promote branching while the third was a standard linear reference material NBS 1475. The analysis was carried out at 160 °C with three PLgel Olexis columns in trichlorobenzene (TCB) with 0.015% butylated hydroxytoluene (BHT) as a stabilizer.

Refractive index and viscometry detectors were employed and the data was analyzed with Cirrus GPC Multi Detector Software using the Universal Calibration approach. Polystyrene standards were used to generate the Universal Calibration and the unbranched sample was used as a linear model in the determination of branching.

Figure 11 shows the molecular weight distributions for the three samples. The black plot is for the unbranched sample. Although there was some overlap, the samples clearly had significantly different molecular weights.

Figure 12 shows the Mark-Houwink plots for the three samples. The uppermost sample is the unbranched material. The other two samples have lower intrinsic viscosities at any given molecular weight, with the unbranched polymer indicating the presence of branching. This can be expressed in terms of g, the branching ratio, defined as follows, where ϵ is a constant:

$$g = \left[\frac{\text{Intrinsic viscosity (branched)}}{\text{Intrinsic viscosity (linear)}} \right]^{1/\epsilon}$$

Conditions

Samples: Polyethylenes
Columns: 3 x PLgel Olexis, 300 x 7.5 mm (Part No. PL1110-6400)
Eluent: TCB + 0.015% BHT
Flow Rate: 1.0 mL/min
Inj Vol: 200 µL
Temp: 160 °C
Detector: PL-GPC 220 + viscometer

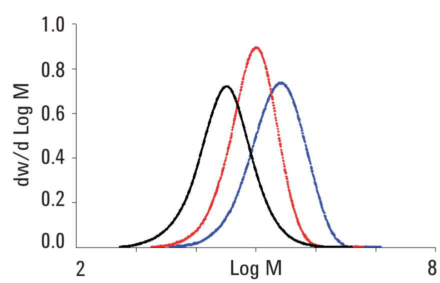

Figure 11. *Molecular weight distribution plots for the three polyethylene samples – the black plot is for the unbranched sample*

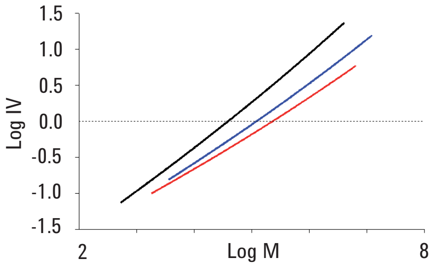

Figure 12. *Mark-Houwink plots for three samples of polyethylene*

The unbranched sample was used as the linear model and so gives a g value of unity (except at high molecular weight due to scatter in the data). The other two samples both exhibit a decrease in g as a function of molecular weight, indicating that as molecular weight increases, the number of branches increases. Based on these calculated g values, a branching number or number of branches per 1,000 carbon atoms can be generated. This is achieved by fitting the data into a model. The Cirrus GPC Multi Detector Software offers a selection of branching models that can be employed in this approach. In this case a model was used that calculates a number-average branching number, assuming a random distribution of branches on the polymer. Figures 13 and 14 show the g plots and branching-number plots obtained for the samples.

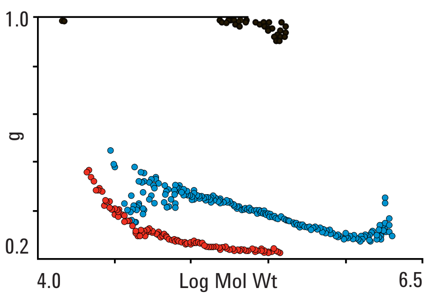

Figure 13. *Branching ratio g plots for the three polyethylene samples – the black plot is the unbranched sample*

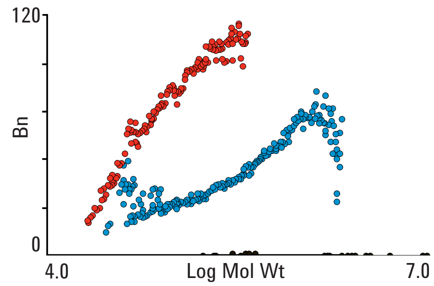

Figure 14. *Calculated branching numbers as a function of molecular weight for three samples of polyethylene – the black plot is the unbranched sample*

The results show that of the two branched samples, the trend in molecular weight distribution does not follow the trend in branching distribution. The sample showing the most branching at any given molecular weight has a lower molecular weight than the second sample. Clearly, understanding both the molecular weight and branching distributions will give an insight into the processibility of the two materials.

Polyphenylene sulfide analysis

Application areas: High performance membranes, felts and insulators

Polyphenylene sulfide (PPS) is an engineering polymer with a rigid backbone of alternating aromatic rings linked by sulfur atoms. It is useful as a structural material due to its high resistance to both chemical and thermal attack, and the material is very stiff, even at high temperatures. PPS is used in a number of applications, including as a filter fabric for coal boilers, in felts used in paper making, in electrical insulation applications and in the manufacture of specialty membranes. PPS is naturally insulating, although the addition of a dopant can be used to make the material semi-conducting.

PPS is particularly difficult to analyze by GPC. The high chemical and thermal resistance of the material means that it is only soluble in specialist solvents such as ortho-chloronaphthalene at elevated temperatures of around 200 °C. The PL-GPC 220 is capable of operation at these temperatures, and the PLgel column material can perform the analysis of PPS.

Conditions

Columns: 3 x PLgel 10 µm MIXED-B, 300 x 7.5 mm (Part No. PL1110-6100)
Eluent: o-chloronaphthalene
Flow Rate: 1.0 mL/min
Temp: 210 °C
Detector: PL-GPC 220

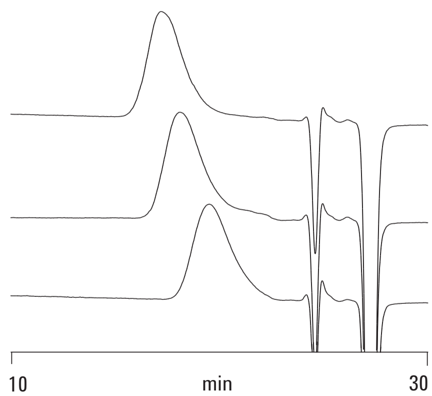

Figure 15. *Overlaid chromatograms of three samples of polyphenylene sulfide*

Ordering information

The following products are featured in this application compendium. For a full list of GPC/SEC part numbers, visit **www.agilent.com/chem/store**

Columns

Description	Part No.
Agilent PLgel 3 µm 100Å, 300 x 7.5 mm	PL1110-6320
Agilent PLgel 5 µm MIXED-D, 300 x 7.5 mm	PL1110-6504
Agilent PLgel 5 µm MIXED-C, 300 x 7.5 mm	PL1110-6500
Agilent PLgel 10 µm MIXED-B, 300 x 7.5 mm	PL1110-6100
Agilent PLgel 20 µm MIXED-A, 300 x 7.5 mm	PL1110-6200
Agilent PLgel 20 µm MIXED-A LS, 300 x 7.5 mm	PL1110-6200LS*
Agilent PLgel Olexis, 300 x 7.5 mm	PL1110-6400
Agilent PL HFIPgel, 300 x 7.5 mm	PL1114-6900HFIP

Standards

Description	Part No.
Agilent PS-H EasiVial 2 mL pre-weighed polystyrene calibration kit	PL2010-0201
Agilent PS-M EasiVial 2 mL pre-weighed polystyrene calibration kit	PL2010-0301
Agilent PS-L EasiVial 2 mL pre-weighed polystyrene calibration kit	PL2010-0401
Agilent EasiCal PS-1 pre-prepared polystyrene kit	PL2010-0501
Agilent EasiCal PS-2 pre-prepared polystyrene kit	PL2010-0601
Agilent PM EasiVial 2 mL pre-weighed polymethylmethacrylate calibration kit	PL2020-0201
Agilent PM EasiVial 4 mL pre-weighed polymethylmethacrylate calibration kit	PL2020-0200

Instruments

Description	Part No.
Agilent PL-GPC 220 Integrated GPC/SEC System	PL0820-0000
Agilent PL-BV 400HT Online Integrated Viscometer	PL0810-3050
Agilent PL-HTLS 15/90 Light Scattering Detector	PL0640-1200
Agilent PL-SP 260VS Sample Preparation System**	

Software

Description	Part No.
Agilent Cirrus GPC Software	PL0570-2000
Agilent Cirrus GPC Multi Detector Software	PL0570-2020

** Low shedding for light scattering applications*
*** Contact your local sales office or distributor for different options*

Companion products

The term engineering polymers includes a wide range of materials, and although gel permeation chromatography is the paramount technique in their analysis, there are other analytical techniques that may be employed. Agilent makes a range of instruments in molecular spectroscopy and X-ray crystallography that can be used for the investigation of these types of material. Agilent's advanced instruments elucidate their characteristics and composition, leading to a better understanding of the behavior and fitness for purpose of these increasingly valuable products.

UV-Vis-NIR spectroscopy

Agilent's Cary range of UV-Vis-NIR spectrophotometers has been synonymous with excellence and high performance for over 60 years. The Cary spectrophotometer series is the standard for researchers wanting to extend the boundaries of spectrophotometric measurement. The range is equally at home in routine laboratories where reliability and ease of use are vital for the quality control of polymers.

The Agilent 600-IR series provides the highest level of sensitivity combined with detailed structural and compositional information for information-rich detection

Fluorescence spectroscopy

Agilent's Cary Eclipse Fluorescence Spectrophotometer offers the high performance you've come to expect from a Cary, at a surprisingly low price. With xenon flash lamp technology, plug-and-identify electronics and feature-packed, intuitive software, the instrument embodies the Agilent and Cary names.

FTIR spectroscopy

The compositional analysis of polymers is made easy with Agilent's FTIR spectrometers and microscopes that provide the ability to extract specific chemical information from extremely small sample areas. Exclusive to Agilent and taking full advantage of our FTIR attenuated total reflection (ATR) imaging technology, the Specac Imaging Golden Gate Diamond ATR accessory also provides the highest quality chemical images that are distortion- and aberration-free with a preserved aspect ratio, while maintaining the Golden Gate's robustness and ease of use.

Raman spectroscopy

Raman spectroscopy delivers qualitative and quantitative information on chemical species that make up engineering polymers. Raman complements IR spectroscopy, particularly for the study of crystalline polymers. Agilent's Synergy FT-Raman module is the most compact FT-Raman accessory on the market, maintaining the versatility required by research spectroscopists.

X-ray crystallography

X-ray crystallography was famously used to decipher the structure of the DNA polymer in the early 1950s. These days, Rosalind Franklin would probably use Agilent's Xcalibur E, the expert diffractometer for the modern chemical crystallography laboratory and the most popular choice for single wavelength, small molecule crystallography.

Other GPC/SEC resources from Agilent

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Publication	Publication number
Biodegradable polymers	5990-6920EN
Polyolefin analysis	5990-6971EN
Elastomers	5990-6866EN
Low molecular weight resins	5990-6845EN
Introduction to GPC/SEC	5990-6969EN
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