

Industrial Polymers Case Study

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The following test was performed:

1. Standardized Gel Permeation Chromatography (GPC)

Objective

The goal of this study was to demonstrate the importance of GPC in the analysis of commercially available industrial polymers. Herein, 6 industrial polymers, namely polyvinyl chloride, polystyrene, polyethylene, polypropylene, polybutene, and polyethylene-polypropylene copolymer were analyzed by standardized Gel Permeation Chromatography (GPC), an analytical service offered at Jordi Labs.

Summary of Results

Six (6) industrial polymers were analyzed by GPC. The results are summarized in **Table 1-Table** 6.

Background

From automotive parts, housewares, and apparel to medical devices and electronic devices, polymers are widely used in industrial applications of all types. The use of polymers is an economically attractive choice over metals, wood, and ceramics throughout the industrial field due to their versatility and the wide variety of physical/chemical useful properties that they offer.

By far, polyolefins are the most widely used polymers in industrial applications. This is due to the fact that polyolefins are derived from relatively inexpensive natural gases such as ethylene, propylene, and butene. The high volume of polyolefins in industrial applications is also attributed to the fact they can resist damage from water, air, grease, and most commonly used cleaning solvents. Moreover, polyolefins can easily be molded into products of different shapes, which makes them perfect candidates for a wide variety of applications.

Industrially, polymers are mainly used for seals, profiles (windows and doors), pipes, cables, floor coverings, insulation, hoses, belts, tanks, membranes and a large number of other products. As technology advances, different types of polymers are also applied in aircraft, aerospace, sports equipments, printed circuit board substrates, 3D printing plastics, holography, special

apparel (bulletproof vests and fire-resistant jackets), and organic polymer flocculants in water purification.

Molecular weight is an important parameter to gauge the functions aforementioned. Gel permeation chromatography (GPC), an analytical technique for the determination of the molecular weight distribution of polymers (**Figure 1**), can be used to study their size-property relationship.



Figure 1. Overlay of industrial polymers in this study

1. PVC (Polyvinyl chloride)

Polyvinyl Chloride (PVC) is one of the most commonly used thermoplastic polymers in the world. PVC is obtained by free radical polymerization of vinyl chloride. Vinyl chloride (CH₂=CHCl) is synthesized by reacting ethylene with oxygen and hydrogen chloride over a copper catalyst.¹



Figure 2. The chemical structure of PVC

PVC is mainly used in the construction trades, where its rigidity and low flammability are useful in pipe, conduit, siding, window frames, and door frames. In combination with a plasticizer (sometimes in concentrations as high as 50 percent), PVC is also used in the fabrication of floor tile, garden hose, imitation leather upholstery, and shower curtains.

Sample Preparation

A PVC sample was dissolved into THF. The resulting solution was agitated overnight at room temperature, yielding a transparent solution. The sample was then analyzed on a **Jordi Resolve DVB Mixed Bed** column without further preparation.

Results

The calculated molecular weight averages (M_n, M_w, M_z) and dispersity values (PDI) are presented in **Table 1**. The resulting weight fraction below 1 kDa is also presented in **Table 1**. The refractive index chromatogram is presented in **Figure 3**.

Table 1

Actual Mw Unknown

Polymer	M _n (Da)	M _w (Da)	M _z (Da)	PDI	Weight % < 1000 Da
PVC (Relative to PS)	45,763	99,347	189,412	2.17	N.D.

¹ https://www.britannica.com/topic/industrial-polymers-468698/Polyvinyl-chloride-PVC



Figure 3. Refractive index (RI) chromatogram of PVC

2. PS (Polystyrene)

Polystyrene (PS) is a synthetic aromatic thermoplastic polymer, that consists of styrene repeat units. In its natural state, polystyrene is a glassy solid at room temperature but melts if heated above about 100 °C.



Figure 4. Chemical structure of PS

Polystyrene is the most employed thermoplastic polymer,² produced on a scale of several million tons annually. Although naturally transparent, PS can be colored using different colorants and thus finds a broad range of applications from food packaging to thermal insulators in buildings. Most specifically, PS is used in the fabrication of containers, lids, bottles, trays, tumblers and more.

Sample Preparation

A polystyrene sample was dissolved into THF. The resulting solution was agitated overnight at room temperature, yielding a transparent solution. The sample was then analyzed using a **Jordi Resolve DVB Mixed Bed** column without further preparation.

Results

The calculated molecular weight averages (M_n, M_w, M_z) and dispersity values (PDI) are presented in **Table 2**. The resulting weight fraction below 1 kDa is also presented in **Table 2**. The refractive index chromatogram is presented in **Figure 5**.

Table 2

Actual Mw 19,920							
Polymer	M _n (Da)	M _w (Da)	M _z (Da)	PDI	Weight % < 1000 Da		
PS (Relative to PS)	19,900	21,407	22,822	1.08	N.D.		

² J. Chem. Educ. <u>2017</u>, 94, 11, 1790-1793



Figure 5. Refractive index (RI) chromatogram of PS

3. PE (Polyethylene)

Polyethylene (PE), the most common plastic used commercially, consisting of a linear chain of methylene repeat units. PE is obtained upon the polymerization of ethylene in presence of coordination catalysts such as metal chlorides or metal oxides. The most commonly used catalyst is titanium(III) chloride, also called the Ziegler-Natta catalyst. PE can also be obtained by radical polymerization of ethylene.³



Figure 6. Chemical structure of PE

Depending on the degree of branching, crystallinity, and molecular weight, polyethylene can be be classified into different types such as ultra-high molecular weight PE (UHMWPE), high density PE (HDPE), low density PE (LDPE) and more, and thus offers a wide variety of mechanical properties.

In 2017, over 100 million tons of polyethyelene resins were produced annually, which consisted of 34% of the whole plastics, market at the time.⁴ Polyethylene is used in almost all types of polymeric materials. In food contact materials, PE is mainly found in packaging materials such as water bottles, plastic bags, trays, jerry cans, in consumer goods such as ice boxes, toys, utensils, in fibers and textiles such as wiring and cables, pipes, steel pipe coatings, and plenty more.

Sample Preparation

A PE sample was dissolved in 1,2,4-trichlorobenzene (TCB) with 0.5 mg/mL butylated hydroxyltoluene (BHT) at 160 °C for 60 minutes, yielding a transparent solution. The sample was then analyzed using a **Jordi Resolve Mixed Bed 13 \mum** column without further preparation.

Results

The calculated molecular weight averages (M_n, M_w, M_z) and dispersity values (PDI) are presented in

Table 3. The resulting weight fraction below 1 kDa is also presented in

³ https://onlinelibrary.wiley.com/doi/abs/10.1002/14356007.a21_487

⁴ http://advances.sciencemag.org/content/3/7/e1700782

Table 3. The refractive index chromatogram is presented in Figure 7.

Polymer	M _n (Da)	M _w (Da)	M _z (Da)	PDI	Weight % < 1000 Da
Polyethylene (GPC-T)	4,584	64,460	431,512	14.060	5.682
1000 PE RI PE RALS					Λ
800 PE Viscosity PS Standard	I RI				
600					
400					
200		\wedge			
0					
-200					
					

Table 3

Actual Mn Unknown

4. PP (Polypropylene)

Polypropylene (PP) is a synthetic aromatic hydrocarbon polymer consisting of propylene repeat units. PP is a partially crystalline and non-polar thermoplastic polymer, with similar properties to PE.



Figure 8. Chemical structure of PP

Similarly to PE, PP is produced at a large scale annually and finds application in a wide variety of materials. PP is used to manufacture piping systems, medical devices, ropes, carpets, food containers, pharmacy prescription bottles, car batteries, and more.

Sample Preparation

A PP sample was dissolved in 1,2,4-trichlorobenzene (TCB) with 0.5 mg/mL butylated hydroxyltoluene (BHT) at 160 °C for 60 minutes, yielding a transparent solution. The sample was then analyzed using a **Jordi Resolve Mixed Bed 13 \mum** column without further preparation.

Results

The calculated molecular weight averages (M_n, M_w, M_z) and dispersity values (PDI) are presented in **Table 4**. The resulting weight fraction below 1 kDa is also presented in **Table 4**. The refractive index chromatogram is presented in **Figure 9**.

Table 4

Actual Mw Unknown

Polymer	M _n (Da)	M _w (Da)	M _z (Da)	PDI	Weight % < 1000 Da
PP (Relative to PS)	55,156	440,560	1,402,000	7.99	N.D.



Figure 9. Refractive index (RI) chromatogram of PP

5. PB (Polybutene)

Polybutene is a linear aliphatic thermoplastic polymer made from a mixture of 1-butene, 2-butene, and isobutylene.



Figure 10. Chemical structure of PB

Polybutene finds application in several industrial products including sealants, adhesives, coatings, polymer modification, tackified polyethylene films, personal care, and emulsions.⁵ Hydrogenated polybutenes are used in a wide variety of cosmetic preparations, such as lipstick and lip gloss. It is used in adhesives owing to its tackiness.⁵

Sample Preparation

A PB sample was dissolved in 1,2,4-trichlorobenzene (TCB) with 0.5 mg/mL butylated hydroxyltoluene (BHT) at 160 °C for 60 minutes, yielding a transparent solution. The sample was then analyzed using a **Jordi Resolve Mixed Bed 13 \mum** column without further preparation.

Results

The calculated molecular weight averages (M_n, M_w, M_z) and dispersity values (PDI) are presented in **Table 5**. The resulting weight fraction below 1 kDa is also presented in **Table 5**. The refractive index chromatogram is presented in **Figure 11**.

⁵ https://www3.epa.gov/pesticides/chem_search/reg_actions/reregistration/fs_PC-011402_1-Jan-95.pdf



Figure 11. Refractive index (RI) chromatogram of PB

Table	5
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Actual Mw Unknown

Polymer	M _n (Da)	M _w (Da)	M _z (Da)	PDI	Weight % < 1000 Da
Polybutene (Relative to PS)	92,992	724,793	2,192,000	7.79	N.D.

6. PE-PP Copolymer

PE-PP Copolymer is a synthetic rubber obtained by copolymerizing ethylene and propylene monomers. There are two major types of PE-PP copolymers with elastic properties: those made with ethylene and propylene alone and those made with small amounts (approximately 5 percent) of a diene (usually ethylidene norbornene or 1,4-hexadiene). The former is known as EPM (ethylene-propylene monomer) and the latter as EPDM (ethylene-propylene-diene monomer).⁶



Figure 12. Chemical structure of PE-PP copolymer

EPDM has an advantage over EPM in that the residual carbon-carbon double bond (the double bond that remains in the diene molecule after polymerization) can be used to crosslink the samples, which makes EPDM much more resistant to degradation by weathering and sunlight.

The principal uses of EPM are in automobile parts and as an impact modifier for polypropylene. EPDM is employed in flexible seals for automobiles, wire and cable insulation, weather stripping, tire sidewalls, hoses, and roofing film.

Sample Preparation

A PE-PP sample was dissolved in 1,2,4-trichlorobenzene (TCB) with 0.5 mg/mL butylated hydroxyltoluene (BHT) at 160 °C for 60 minutes, yielding a transparent solution. The sample was then analyzed using a **Jordi Resolve Mixed Bed 13 \mum** column without further preparation.

Results

The calculated molecular weight averages (M_n, M_w, M_z) and dispersity values (PDI) are presented in **Table 6**. The resulting weight fraction below 1 kDa is also presented in **Table 6**. The refractive index chromatogram is presented in **Figure 13**.

⁶ https://www.britannica.com/science/ethylene-propylene-copolymer



Figure 13. Refractive index (RI) chromatogram of PE-PP copolymer

Table 6

Actual Mw Unknown

Polymer	M _n (Da)	M _w (Da)	M _z (Da)	PDI	Weight % < 1000 Da
PE-PP copolymer (Relative to PS)	122,544	355,150	1,064,000	2.90	N.D.

Closing Comments

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Jordi Labs specializes in polymer analysis and has more than 35 years' experience performing regulatory, quality control and failure testing. We are one of the few labs in the United States specialized in this type of testing. We will work closely with you to help explain your test results and complete your project goals. We appreciate your business and are looking forward to speaking with you concerning these results.

Sincerely,

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