Fullerene Analysis using Agilent PLgel Columns and Gel Permeation Chromatography

Application Note

Materials Testing and Research, Polymers

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Introduction

Fullerenes are normally prepared by evaporating graphite rods in arc reactors under a helium atmosphere to produce soot that contains C_{60}, C_{70}, higher fullerenes, and uncharacterized insoluble material. The soluble fractions may be extracted using a suitable solvent and further purification may be carried out by gel permeation chromatography (GPC). Low pore size Agilent PLgel 50Å GPC columns are most appropriate for these applications. As an analytical tool, high efficiency columns can be used to screen fullerene compounds. The behavior of preparative separations can be predicted by running an analytical separation using the same column packing.

Analysis of Fullerene

One of the difficulties in fullerene chromatography is the relatively poor solubility of the compounds. However, toluene exhibits favorable properties as a GPC solvent since it provides reasonable solubility (6 to 8 mg/mL), good chromatographic selectivity, and is amenable to solvent removal and recovery by distillation in preparative separations.

Figure 1 illustrates the separation achieved for a material whose basic structure was believed to be that indicated in Figure 2.

Verified for Agilent
1260 Infinity
GPC/SEC System

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Conditions for Figure 1

Columns	2 × Agilent PLgel 5 µm 50Å, 7.5 × 300 mm (p/n PL1110-6515)
Eluent	Toluene
Flow rate	1.0 mL/min
Loading	2 mg/mL, 200 µL
Detector	RI
System	Agilent PL-GPC 50

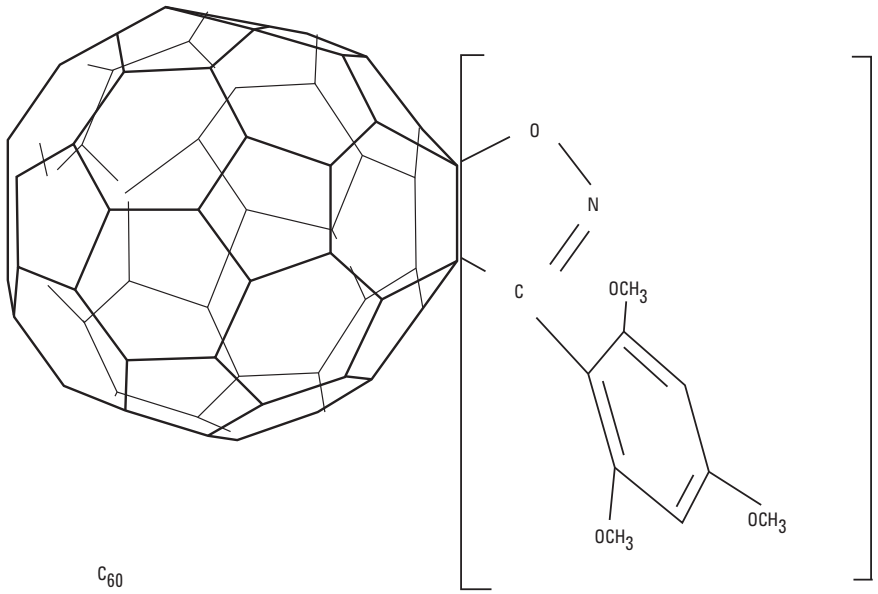

Figure 1. Separation of a fullerene on an Agilent PLgel two-column set.

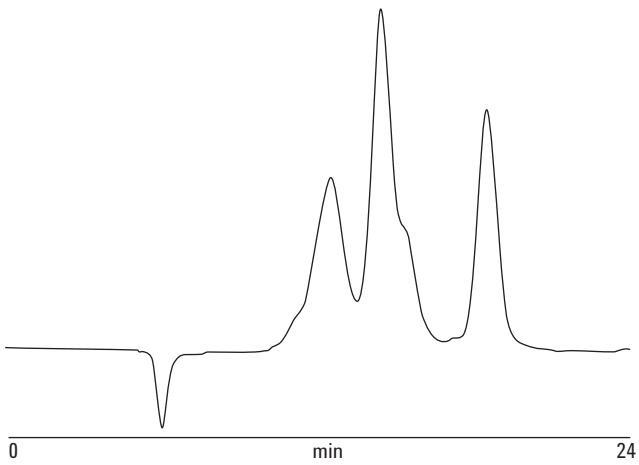

Figure 2. Suspected structure of the fullerene under investigation.

In preparative work, columns packed with larger particle size packings are preferred for increased loading. Preparative column separations can easily be predicted by running samples on analytical columns packed with the same type of beads. Figure 3 illustrates this approach; if this separation was scaled up to a preparative system it would require 25-mm id columns with a subsequent tenfold increase in flow rate and loading.

Conditions for Figure 3

Columns	4 × Agilent PLgel 10 µm 50Å, 7.5 × 300 mm (p/n PL1110-6115)
Eluent	Toluene
Flow rate	1.0 mL/min
Loading	2 mg/mL, 200 µL
Detector	RI
System	PL-GPC 50

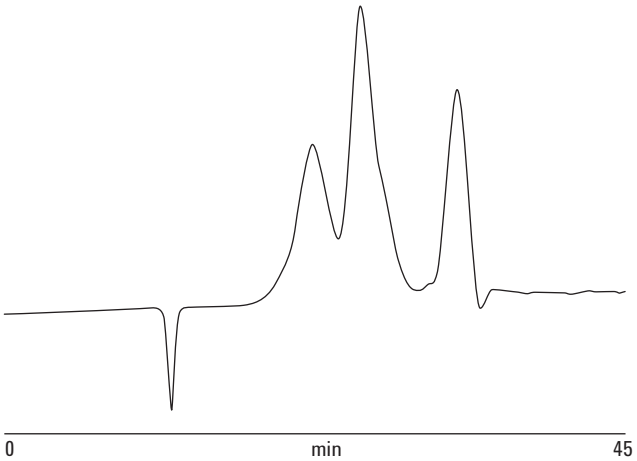

Figure 3. Separation of fullerene using an Agilent PLgel 10 µm analytical packing to predict a preparative scale separation using the same packing.

Conclusion

Soluble fullerene fractions extracted using a suitable solvent can be further purified by gel permeation chromatography using low pore size Agilent PLgel 5 µm columns. The behavior of preparative separations can be predicted by first running an analytical separation with larger pore sized Agilent PLgel 10 µm columns, using the same column packing.

For More Information

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