WYATT TECHNOLOGY

APPLICATION NOTE

AN1006: Polyurethane – absolute molar mass by SEC-MALS

Introduction

Polyurethane is a common polymer which is sometimes difficult to analyze. Polyurethanes are used especially in flexible and rigid foams, elastomers and resins and their molecular weight properties contribute significantly to their performance characteristics.

Polyurethanes are typically analyzed by gel-permeation chromatography (GPC, or size-exclusion chromatography, SEC) using polymer standards and extensive column calibration. Besides being tedious, column calibration means that the resulting answers will be relative and not absolute, relying on the assumption that the reference standards have the same conformation, density and column interactions as the analyte of interest.

A far more effective means of characterizing polyurethanes involves adding multi-angle light scattering (MALS) to SEC, creating a SEC-MALS system which eliminates the need for column calibration and reference standards.[1] SEC-MALS analyses derive from first principles; they are absolute and do not depend on such assumptions.

Materials and Methods

A polyurethane sample was dissolved in DMF with 0.01 M LiBr. The specific refractive index increment, dn/dc, was measured off-line by injecting aliquots at a series of carefully prepared concentrations into an Optilab® differential refractometer. The Optilab uses a light source at the same wavelength as the DAWN® multi-angle light scattering detector, in order for the measured dn/dc value to provide maximal accuracy in concentration and MALS analyses.

An aliquot was then injected onto the SEC column at a flow rate of 0.485 mL/min. The eluting fractions were characterized by the DAWN and Optilab for determination of the molar mass of each one-second slice.

Results and Discussion

The following results—courtesy of Kolon Corp., Korea—display the absolute molar mass at each slice in the light scattering chromatogram (Figure 1) and the conversion to a differential distribution (Figure 2). The logarithmic molar mass-elution volume relationship displayed provides clear indication that the separation is ideal and no unexpected column interactions occur.

The results are further analyzed to produce the absolute weight-average molar mass M_w, other molecular weight moments of the peak and the polydispersity M_w/M_n. These are shown in Table 1 as are the uncertainties of each of the calculated quantities.

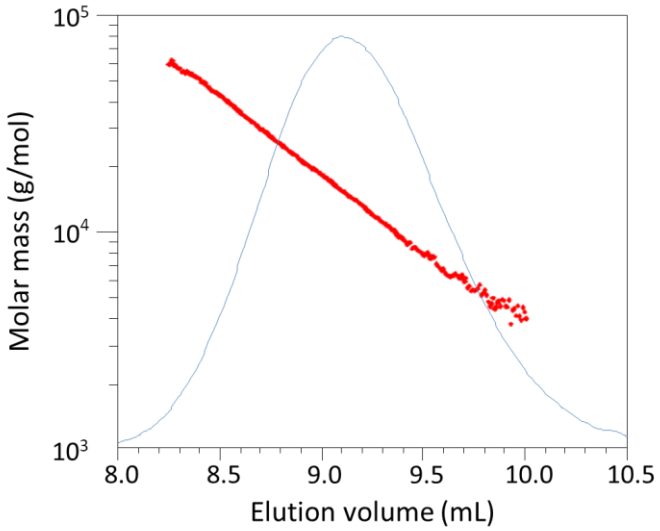

Figure 1. A plot of the molecular weight vs. volume for the polyurethane sample overlaid upon the trace from the 90° light-scattering detector.

Table 1. Molar mass and polydispersity results from SEC-MALS.

Property	Value
Peak range	8.25 - 10.00 mL
Injected mass	2.058 x 10^{-4} g
M_n - number-average MW	(1.36±0.03) x 10^4 g
M_w – weight-average MW	(2.02±0.02) x 10^4 g
M_z – z-average MW	(2.87±0.05) x 10^4 g
Polydispersity M_w/M_n	1.49±0.04

Conclusions

While standard GPC is considered to be the primary technique for determination of molar mass distributions, only SEC-MALS provides unbiased and absolute molar mass measurements. Simply adding a MALS instrument to an existing GPC system reveals new horizons in absolute polymer characterization.

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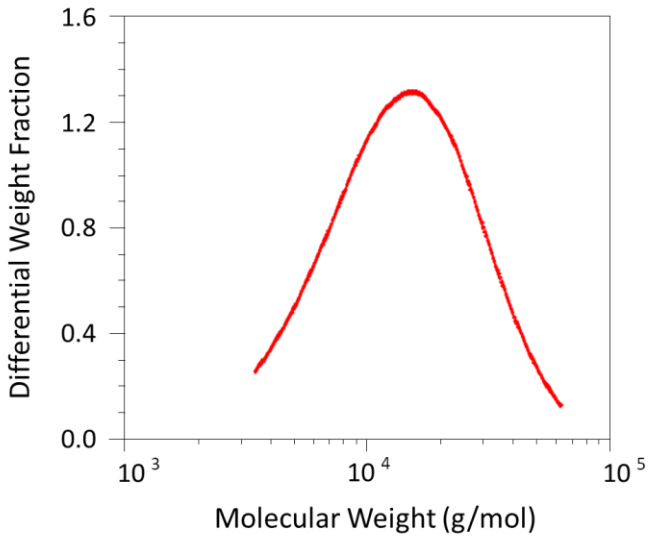
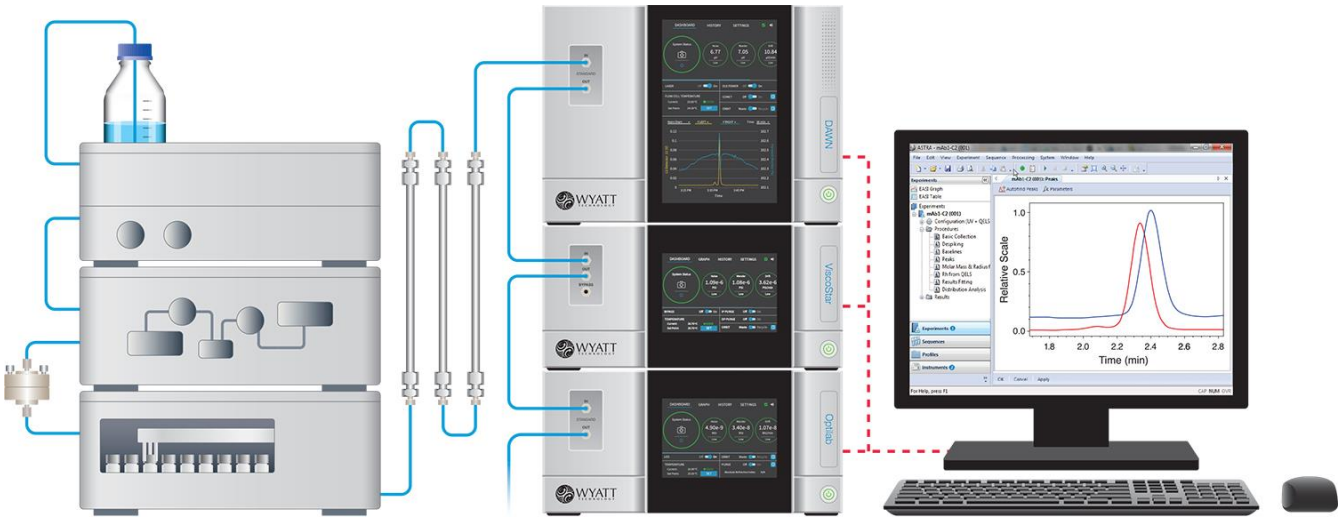


Figure 2. The differential molecular weight distribution of this polyurethane ranges from about 4 kDa to more than 60 kDa.

References

1. Wyatt, P. J. Light scattering and the absolute characterization of macromolecules. *Anal. Chim. Acta* **272**, (1993).



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