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Molecular Weight Analysis of Synthetic Polymers and Simultaneous Analysis of Polymer Additives

Simultaneous Analysis of Polymer Additives by GPC (HPLC)

Measuring the molecular weight distribution of polymers is a branch of HPLC analysis performed in size exclusion mode that has long been called **gel permeation chromatography (GPC)**. The recent demand for improved throughput by increasing analysis speed is considered important even for GPC, which has an established analytical procedure. Presented here is an example of using overlapped injection on a normal column to improve efficiency and, simultaneously, perform a quantitative analysis of polymer additives.

A weight-responsive refractive index detector (RID) is often used in GPC analysis when determining the mean molecular weight and polydispersity of polymer compounds. UV detectors are often used when analyzing additives with antioxidant properties because many of the compounds have double bonds. Fig. 32 shows the chromatogram obtained from GPC analysis of the polystyrene sample and three additives used in this investigation. Table 12 shows results for the polystyrene sample.

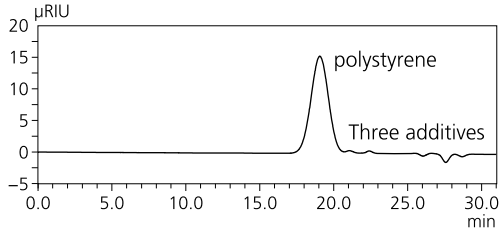

Fig. 32 Chromatogram of Polystyrene and Additives

Table 12 Analytical Results for the Polystyrene Sample (n = 6)

	Number average molecular weight Mn	Weight average molecular weight Mw	Polydispersity Mw/Mn
Polystyrene	2.63×10^4	4.89×10^4	1.86
%RSD	1.41	0.89	0.52

When multiple additives are present, achieving complete separation of small molecule additives from each other can be difficult even on columns with small exclusion limits, and an accurate quantitative analysis is almost always difficult to achieve. Therefore, a photodiode-array (PDA) detector that also provides spectral information was used as the UV detector, and a peak deconvolution function was used to improve the separation of unresolved additives. A LabSolutions (workstation) feature called **i-PDeA II** separates unresolved peaks based on three-dimensional spectral information obtained with a PDA. Fig. 33 shows the chromatogram of the peak detected at UV 240 nm by the PDA and a superimposed chromatogram of each individual component obtained with the peak deconvolution function. Only two peaks could be detected by UV detection alone, but three peaks were identified after processing with i-PDeA II. Furthermore, the peak areas obtained with the deconvolution function represent the contribution of each component in the original unresolved data and can be used without processing for quantitative calculations.

Calibration curves were made for the three additives in the range of 0.01 to 0.1 % (w/v) and used to calculate the quantities of additives added to the polystyrene sample. Table 13 shows the linearity of each calibration curve and the quantitative results of repeat analysis (six times). Quantitative calculations by i-PDeA II, which compensates for the separation performance of GPC columns in the low molecular weight range, demonstrated how GPC can potentially provide value-added, high-throughput analysis.

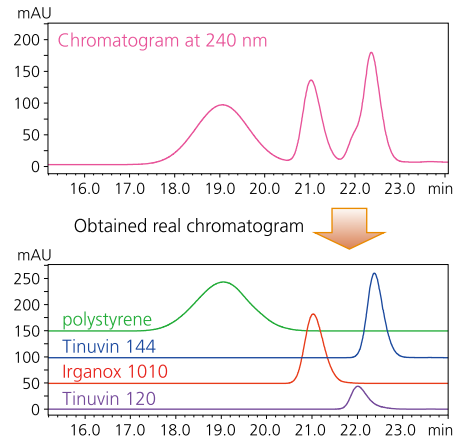

Fig. 33 Deconvolution Results for Three Additives (PDA)

Table 13 Analytical Results for Additives in a Polystyrene Sample (n = 6)

Additive	Irganox 1010	Tinuvin 144	Tinuvin 120
Linearity of calibration curve (r^2)	0.999	0.995	0.998
Determined content (mg/g)	49.2	23.1	27.4
%RSD	1.28	1.93	1.47