High Sensitivity Analysis of Natural Rubber by GPC with Evaporative Light Scattering Detection

Application Note

Authors

Graham Cleaver
Agilent Technologies, Inc.

Introduction

Solutions of natural rubber samples are generally very difficult to prepare for GPC due to the fact that the polymer contains relatively high levels of 'gel' that is partially crosslinked. Normally, an aliquot of the eluent is added to the weighed sample. This is allowed to swell and dissolve overnight, and then the gel material is filtered out (0.5 µm) prior to GPC analysis.

In this case, the actual polymer concentration can be significantly lower than the original concentration prepared depending on the gel content of the sample, and therefore detector response, usually RI, tends to be quite poor. The Agilent ELSD exhibits significantly increased sensitivity compared to an RI and gives much greater response for this application. In addition, RI baseline drift, which commonly occurs, is very much emphasized when the actual peak response is so small. The Agilent ELSD always gives a flat baseline which, together with the improved response, makes baseline and peak setting much more reliable for GPC calculations.

RI is also sensitive to system peaks around total permeation that usually occur even when samples are prepared in an aliquot of the eluent. These system peaks can interfere with low molecular weight components that are commonly found in natural rubber samples. With the Agilent ELSD system peaks are eliminated due to evaporation, leaving unadulterated sample peaks in the additives region.

The PLgel 10 µm MIXED-B columns, with their high efficiency (>35,000 plates/meter) and broad resolving molecular weight range (up to 10,000,000 daltons relative to polystyrene), are the columns of choice for high molecular weight polymers and demanding eluents.

Separation of natural rubber reveals that the combination of PLgel 10 µm MIXED-B columns with the Agilent ELSD comprises a highly sensitive system for the discrimination of additives.

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Instrumentation
Columns: 3 x PLgel 10 µm MIXED-B, 300 x 7.5 mm (p/n PL1110-1120)
Detector: Agilent ELSD (neb=50 °C, evap=90 °C, gas=1.0 SLM)

Materials and Reagents
Eluent: Toluene

Conditions
Flow Rate: 1.0 mL/min

Results and Discussion

Figure 1 shows chromatograms of two samples of natural rubber on RI and Agilent ELSD detectors. Figure 2 is a magnified view of the additive area revealing the unadulterated peaks in this region of interest.

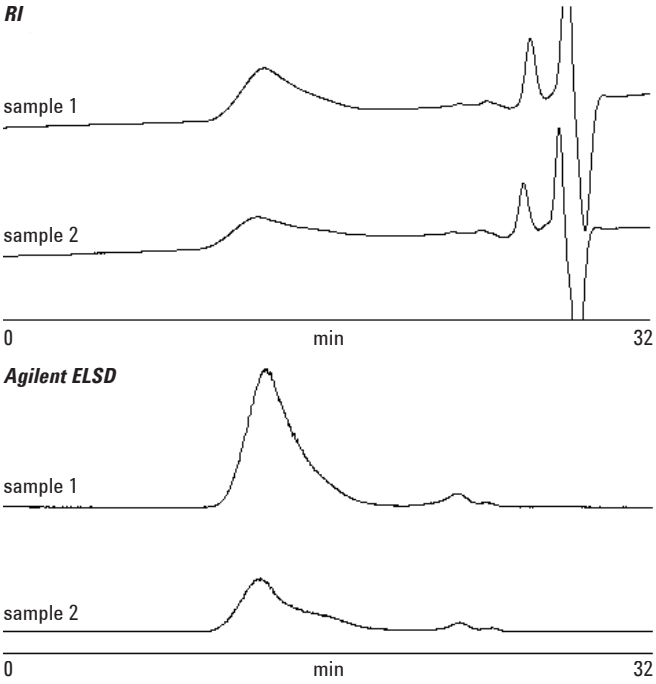

Figure 1. Stable base line and no interference from system peaks using the Agilent ELSD (below) compared to RI detection (above).

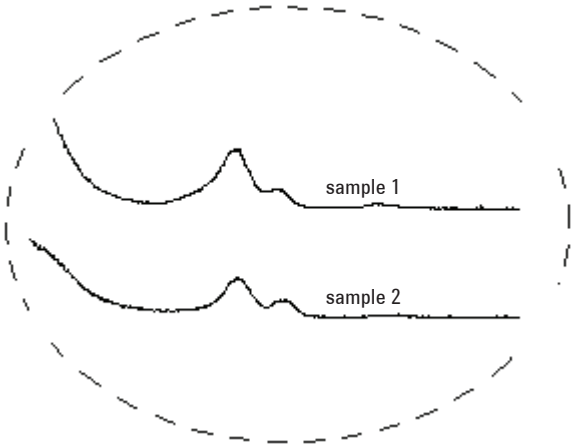

Figure 2. Magnified view of the Agilent ELSD plots showing the additive region.

Conclusion

PLgel 10 µm MIXED-B columns and the Agilent ELSD provide an excellent combination for the molecular weight determination of natural rubbers because of the system's high sensitivity, very low signal to noise ratios and excellent base line stability.

Mixed pore size PLgel columns offer high resolution over a specific molecular weight range. The robust design of the Agilent ELSD allows the nebulizer and evaporator to operate at very high temperatures, efficiently handling the high boiling point solvents that other ELSDs simply cannot manage.

PLgel columns and the Agilent ELSD are well suited to the separation of compounds that have no chromophores under isocratic or gradient conditions.

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