Optimal Analysis of Epoxy Resins by GPC with ELSD

Application Note

Materials, Testing and Research

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Introduction

Epoxy resins are used widely in industry because of their strong adhesive properties, chemical resistance, mechanical toughness and high electrical insulation. Consequently, they are employed in adhesives, stabilizers, sealants, varnishes and paints. There are various types of epoxy resins that correspond to differences in molecular weight distribution and oligomeric profile. The distribution of oligomers in epoxy resins dictates their physical nature and so the characterization of epoxy resins is highly important in their quality and process control. The analysis of low molecular weight epoxy resins can be achieved by GPC or HPLC using RI or UV detection. RI detection is typically used for isocratic separations, whereas for gradient analysis UV detection is usually favored. While these detection methods are suitable for simple epoxy resin determination, they are insufficient for analyzing complex mixtures of epoxy resins and non-UV absorbing impurities. Evaporative light scattering detection (ELSD) offers significant benefits over RI and UV, as it is not dependent on the optical properties of the compound. ELSD can detect any compound that is less volatile than the mobile phase; it is compatible with a wide range of solvents, is insensitive to solvent gradients and displays excellent baseline stability. A PLgel MIXED column of appropriate pore size is the optimum choice for polydisperse compounds. In this case, the preferred option is a PLgel 3 µm MIXED-E column, an ultra high efficiency column for low molecular weight compounds. The benefits of combining ELSD with a PLgel 3 µm MIXED-E column are highlighted in the analysis of epoxy resins, such as epikotes.

Verified for Agilent
1260 Infinity ELSD
1290 Infinity II ELSD

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Instrumentation

Columns: 2 x PLgel 3 µm MIXED-E, 300 x 7.5 mm (p/n PL1110-6300)
Detection: Agilent 380-ELSD (neb=50 °C, evap=90 °C, gas=0.8 SLM)

Materials and Reagents

Eluent: THF

Sample Preparation

Sample: Epikote 1001 (Hexion Specialty Chemicals, Columbus OH, USA)

Conditions

To achieve optimum sensitivity on the Agilent 380-ELSD, the evaporator temperature was set to 90 °C as the analyte is non-volatile. In addition, the gas flow was turned off, because the mobile phase of THF was easily removed at this temperature.

Flow Rate: 1.0 mL/min
Injection Volume: 10 µL

Results and Discussion

The low dispersion of the Agilent 380-ELSD produced peak shapes comparable to those obtained by UV, and more responsive than RI detection, as shown in Figure 1.

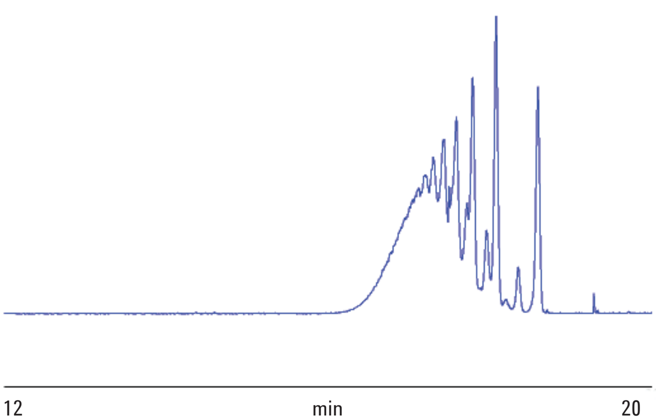

Figure 1. Chromatogram showing the detection of the main sample peak and two contaminants.

Conclusion

The Agilent 380-ELSD was successfully used to elucidate molecular weight distribution of an epoxy resin. Coupled with a PLgel 3 µm MIXED-E column, optimized for the rapid analysis of low molecular weight materials below 30,000 MW, the ELS detector was more responsive than RI because it is independent from the optical characteristics of the analyte. This system is also suited to the analysis of prepolymers, oils and additives.

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