

Superior Analysis of Alkylketene Dimers by GPC with Evaporative Light Scattering Detection

Application Note

Authors

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Introduction

Alkylketene dimers (AKD) are widely used as sizing agents in paper production. AKD is not a pure material because it is prepared from commercial grades of stearic acid that contain a range of fatty acid impurities with chain lengths varying from C14 to C20, hence the pluralization to dimers.

Gel permeation chromatography is used for both qualitative and quantitative analysis of AKD. The level of AKD present in the sizing fluid can be monitored before, during and after the sizing procedure. The Agilent ELSD evaporative light scattering detector is an excellent detector for AKD since it provides the benefit of significantly improved signal to noise ratio as the detector response is almost independent of dn/dc effects.

The PLgel 3 μ m MIXED-E columns, with their high efficiency (>80,000 plates/meter) and broad resolving molecular weight range (up to 30,000 daltons relative to polystyrene), are the columns of choice for low molecular weight prepolymers.

Coupling PLgel MIXED-E columns with the Agilent ELSD provides an ideal combination for the analysis of alkylketene dimers.



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Instrumentation

Columns: 2 x PLgel 3 µm MIXED-E, 300 x 7.5 mm (p/n PL1110-6300)
Detection: Agilent ELSD (neb=40 °C, evap=90 °C, gas=1.5 SLM)

Materials and Reagents

Eluent: THF

Conditions

Flow Rate: 1.0 mL/min

Results and Discussion

As the dimer species is predominant in AKD, quantitation via peak area measurements is performed (Figure 1). AKD can be present in variable quantities and the high sensitivity of the Agilent ELSD facilitates quantitation even at very low concentration.

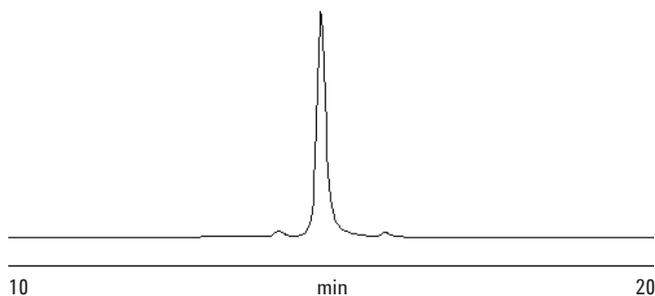


Figure 1. Typical full scale GPC chromatogram of a sample of AKD using Agilent evaporative light scattering detection.

The effectiveness of AKD as a sizing agent is related to the distribution of oligomers, which form by condensation reactions after hydrolysis in the sizing fluid. GPC separates according to molecular size in solution and is therefore ideal for monitoring the extent of the reaction. High resolution GPC columns permit separation of individual AKD oligomers as illustrated in Figure 2.

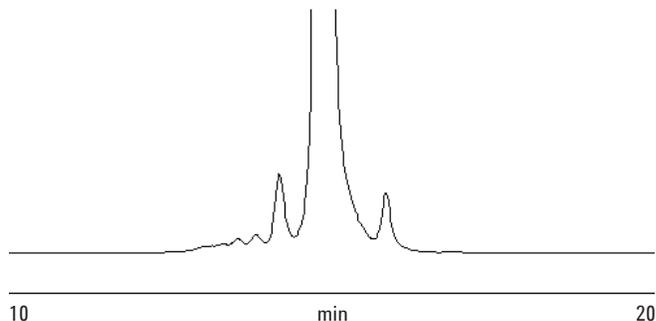


Figure 2. A close up of the region around the main AKD peak revealing the presence of smaller quantities of other oligomers.

The high sensitivity of the Agilent ELSD permits detection of the low level components and would permit a quantitative analysis of AKD products in extracts of sized paper.

Conclusion

PLgel columns and Agilent ELSD are ideal for the determination of AKD because of the system's very low signal to noise ratios and excellent baseline 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 Agilent ELSD are well suited to the separation of compounds that have no chromophores, under isocratic or gradient conditions.

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