Single-stage Extract Clean-up in Pesticides' Determination Using Agilent EnviroPrep

Application Note

Food Testing and Agriculture

Author

Graham Cleaver

Agilent Technologies, Inc.

Introduction

High performance gel permeation chromatography (GPC) provides a simple, one stage clean-up procedure for the determination of pesticides in a variety of organic matrices, such as soil and animal tissue. The matrix is extracted and the higher molecular weight fractions such as lipids, polymers, natural resins and dispersed high molecular weight components are easily eliminated by GPC. Since these species are relatively non-volatile, their removal from the extract prior to GC/MS analysis of pesticides and other pollutants extends the lifetime of the GC column, and improves chromatographic efficiency.

An application of preparative GPC in the clean-up of soil extracts is described in EPA Method SW846-3640 and is the US EPA CLP Statement of Work for Organics Analysis Document Number OLM01.0. Preparative GPC is preferred since higher sample loadings and fraction yields can be obtained, which is particularly useful for looking at low levels of pollutants. In addition, analytical GPC methods have also been successfully applied [1], with the advantage of reduced clean-up time and solvent consumption.

Owing to their relatively small pore size (100Å) and low exclusion limit (4,000 g/mol), Agilent EnviroPrep GPC columns are ideal for this type of preparative application, where they offer high resolution and high loading (>10 mg on-column loading of the components in this analysis) through optimization of the particle size distribution. As a means of evaluating the performance of the GPC column and system, a separation of a test solution is recommended.

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Method and Results

Although this analysis uses dichloromethane, rapid, simple GPC clean-up of this type can also be performed using non-chlorinated eluents such as ethyl acetate/cyclohexane mixture (1:1). The chromatogram in Figure 1 shows that good separation of the principle components in the mixture was achieved.

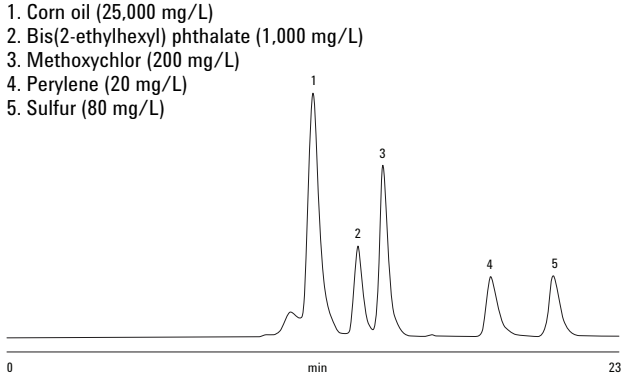

Figure 1. Good separation of a five-component mix on a two-column set of EnviroPrep columns. The separation demonstrates that relatively high molecular weight components can be readily resolved from the low molecular weight compounds of interest.

Conditions

Column	2 x EnviroPrep, 300 x 25 mm (p/n PL1210-6120EPA)
Eluent	Dichloromethane
Inj Vol	2 mL
Flow Rate	10 mL/min
Detector	Agilent 1260 Infinity Variable Wavelength Detector VL, 254 nm
System	Agilent 1260 Infinity Isocratic Pump and Manual Injector

Conclusion

The criterion for the test separation is that each of the peaks can be resolved in the sample. With EnviroPrep, sharp peaks are obtained with clear separation, illustrating the value of the column for applications covered by the EPA method. For sample clean-up in minimal time with maximum throughput, highly mechanically stable EnviroPrep columns can be operated up to 2,200 psi (150 bar) with eluent flows of up to 10 mL/min. In addition, for maximum flexibility, the columns can be used with any liquid chromatography system capable of isocratic flow at the required flow rates.

Reference

1. A. Khoshaband and R. Teasdale. (1994) Journal of Chromatography, 660, 195-198.

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