Gel Permeation Chromatography of Phthalate in Walnut Oil Using Agilent EcoSpheres

Application Note

Food Testing and Agriculture

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Introduction

Walnut oil from the common walnut, Juglans nigra L., Juglandaceae, is high-quality oil known for its delicate, nutty flavor and therefore used in cooking. The oil is produced mainly in France and is an expensive commodity that can become rancid if incorrectly stored. As part of a production process, walnut oil was to be packaged in plastic bottles manufactured from polyvinyl chloride (PVC). A stiff plastic, PVC is made flexible by the addition of plasticizers, typically phthalates, which help the polymer chains slide past each other. However, phthalates have been implicated as a potential health risk, raising concerns about phthalates leaching into foodstuffs.

This Application Note shows the separation of a phthalate additive from a sample of walnut oil as part of a quality control procedure, to isolate and measure the quantity and type of phthalate. To perform the separation, a 450 x 25 mm glass column was packed with Agilent EcoSpheres swollen in tetrahydrofuran.

EcoSpheres beads are designed for the economic, low pressure separations of pesticides, chlorinated hydrocarbons and other small molecules from high molecular weight organic matrices.

Verified for Agilent
1260 Infinity
GPC/SEC System

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Method and Results

Conditions

Column	EcoSpheres glass column, 450 x 25 mm (EcoSpheres loose media (100 g) p/n PL1460-4M03; complete glass column p/n PL1310-0054)
Sample	Walnut oil spiked with phthalate
System	Agilent 1260 Infinity Isocratic Pump and Manual Injector
Eluent	Tetrahydrofuran
Flow Rate	5 mL/min
Detector	Agilent Infinity Variable Wavelength Detector VL, 254 nm
Data collection/ analysis	Cirrus GPC software and PL DataSteam data capture unit

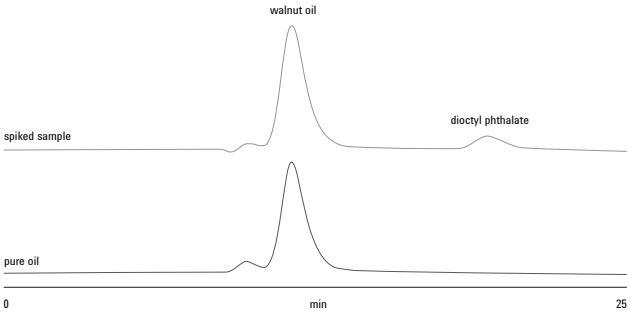

Figure 1. Overlaid chromatogram of samples of spiked and un-spiked walnut oil produced by gel permeation chromatography with EcoSpheres microporous media. Clearly the phthalate is well resolved from the oil, allowing the small molecule to be isolated for further testing.

Conclusions

A GPC set up with EcoSpheres media is an easy and simple method for the analysis of food samples such as walnut oil. In this example, a phthalate was detected, suggesting such a system would be useful in the quality control of walnut oil packed in PVC containers.

For faster analysis times, the large particle size of EcoSpheres beads allows a 25 mm id glass column to be used at a flow rate of up to 10 mL/min, up to a maximum of 300 psi (21 bar). For maximum flexibility, EcoSpheres can be used with any liquid chromatography system capable of isocratic flow at the required flow rates. In addition, the pore size of EcoSpheres has been selected to maximize the separation of low molecular weight species such as phthalate from higher molecular weight interferences, which are excluded from the pores of the material.

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