Fast Separation of Food Preservatives Using an Agilent Poroshell 120 EC-C18 Column

Application Note

Food Testing & Agriculture

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Abstract

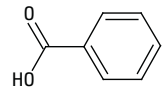
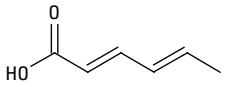
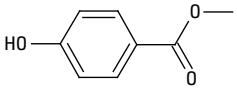
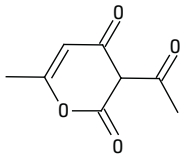
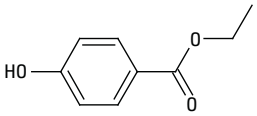
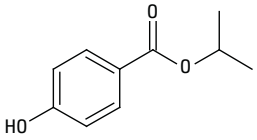
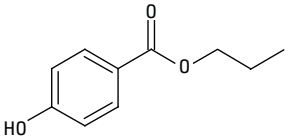
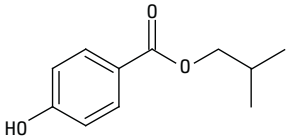
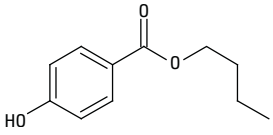
A UHPLC method was developed for the simultaneous determination of the nine preservatives most often used in food and beverages. An Agilent Poroshell 120 EC-C18 column was used for the separation with a gradient method and acetate ammonium buffer: acetonitrile mobile phase. This method gave a rapid separation of the preservatives in 9 minutes. It is suitable for many food and beverage samples and was applied here to the analysis of these preservatives in a cake sample.

Introduction

Preservatives are very popular in the food and cosmetics industries because they prevent these products from degrading within the warranty time. However, preservatives are strictly regulated because their overuse can cause some health problems in humans. For example, some preservatives can accumulate in the human body and negatively influence the metabolism process. Today's trends in food and cosmetics increasingly emphasize the concepts of health and green issues. This means use of safer raw materials as well as fewer preservatives and control of preservatives within a safe limit. Many regulations made by the US Food and Drug Administration (FDA), the European Union (EU), and others set concentration limits on the preservatives in food.

Table 1 lists preservatives commonly used in food. The HPLC method has been the most popular way to analyze the preservatives in food samples. A previous application note described a method developed using a traditional Agilent J&W HC-C18 (2), 4.6 × 250 mm, 5 µm column (p/n 588905-902) that simultaneously separated nine preservatives in 30 minutes [1]. This application note focused on developing a method for rapidly separating the nine compounds and a food sample using a Poroshell 120 EC-C18 column.

Table 1. Preservatives used in this study.

Peak No.	Name	CAS	Structure
1	Benzoic acid	65-85-0	
2	Sorbic acid	110-44-1	
3	Methylparaben	99-76-3	
4	Dehydroacetic acid (DHA)	520-45-6	
5	Ethylparaben	120-47-8	
6	Isopropylparaben	4191-73-5	
7	*n*-Propylparaben	194-43-3	
8	Isobutylparaben	4247-02-3	
9	*n*-Butylparaben	94-26-8	

Materials and Methods

HPLC analysis was performed with the Agilent 1290 Infinity LC System, including an Agilent 1290 Infinity Binary Pump (G4220A), an Agilent 1290 Infinity Autosampler (G4226A), an Agilent 1290 Infinity Thermostatted Column Compartment (G1316C), and an Agilent 1290 Infinity Diode Array Detector (G4212A).

Conditions

Column:	Agilent Poroshell 120 EC-C18, 3.0 × 100 mm, 2.7 µm (p/n 695975-302)	
Eluent:	A, 10 mM acetate; B, methanol	
Injection volume:	1 µL	
Flow rate:	0.6 mL/min	
Gradient:	Time (min)	% B
	0	20
	10	50
Stop time:	10 min, post run 2 min	
Temperature:	30 °C	
Detector:	UV, 230 and 260 nm	

The standard solution was prepared in 20% methanol:80% water at 0.1 mg/mL individually. The cake sample was prepared as follows. Weigh 5 g of sample, add 40 mL water and a drop of ammonium hydroxide, and extract in sonicator at 60 to 80 °C for 30 minutes. Add methanol to 50 mL, and filter using Agilent 0.2 µm regenerated cellulose membrane filters (p/n 5064-8222) before injection.

Results and Discussion

The previous method was developed on an HC-C18 (2), 4.6 × 250 mm, 5 µm column that separated nine preservatives compounds to baseline in 30 minutes. The original method was transferred to a Poroshell 120 EC-C18, 3.0 mm × 100 mm, 2.7 µm column.

Poroshell 120 columns are packed with superficially porous materials with a particle size of 2.7 µm. Compared to traditional totally porous particle columns, the superficially porous particles produce a fast separation with no compromise in performance due to fast diffusion in the particles. The Poroshell 120, 2.7 µm particles have a Van-Deemter curve similar to columns with sub-2.7 µm totally porous materials. The performance of the Poroshell 120 column does not decrease at high flow rate.

A 3.0 mm id column was used for this separation at a flow rate of 0.6 mL/min, which is approximately 1.5 times of normal flow rate of 0.4 to 0.5 mL/min typically used with this column id. The gradient was optimized according to the column configurations, and nine components were separated completely in 10 minutes on the Poroshell 120 EC-C18 column (Figure 1). The analysis time was shortened from 30 minutes to 10 minutes. Both 230 nm and 260 nm UV wavelength were applied for the detection, because benzoic acid and dehydroacetic acid have a maximum absorption of 230 nm while others have absorptions at 260 nm.

These preservatives are commonly used in many beverages and food, such as fruit-flavored drinks, cakes, candy, and so on. A sample of cake was analyzed, and Figure 2 shows the chromatogram of the sample. Based on the standard, only the dehydroacetic acid was found in the sample at a UV wavelength of 230 nm. The amount could be measured given the standard's concentration.

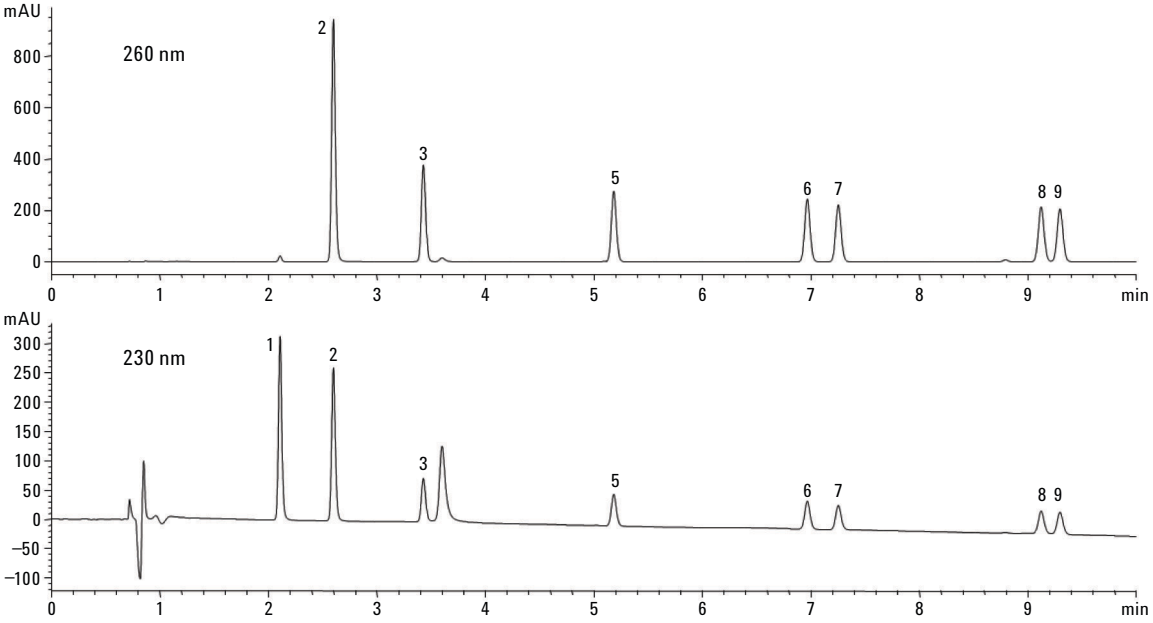


Figure 1. Separation of nine preservative standards using an Agilent Poroshell 120 EC-C18 with UV detection at 230 nm and 260 nm.

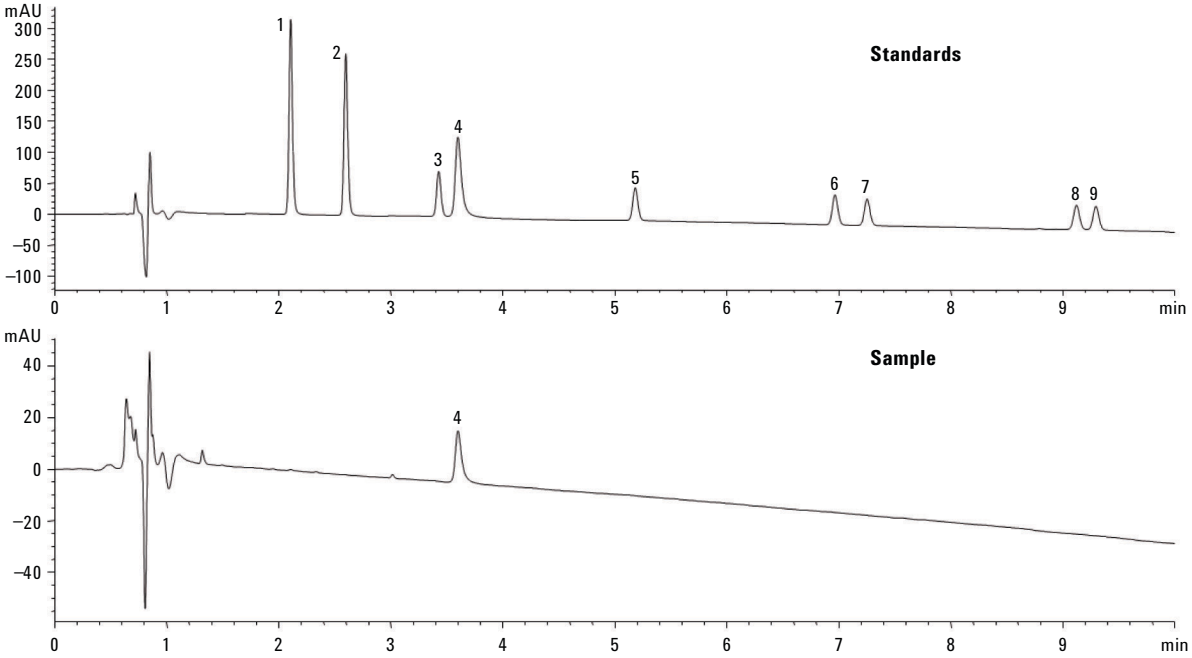


Figure 2. Separation of preservatives in cake using an Agilent Poroshell 120 EC-C18 column with UV detection at 230 nm.

Conclusions

Using a gradient method, preservatives can be rapidly separated on the Agilent Poroshell 120 column. This method allows rapid separation and screening for preservatives in food and cosmetics that can be a routine analysis for product quality control.

For More Information

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Reference

1. Rongjie Fu, Zhixiu Xu. "Analysis of preservatives in food and cosmetics with the Agilent 1120 Compact LC system". Application Note, Agilent Technologies, Inc., Publication Number 5989-8960EN (2010).

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