

## SPME Reduces Extraction Time in HPLC Analyses of Food Antioxidants and Preservatives

*In the few years since its introduction, solid phase microextraction (SPME) has become established as a practical alternative for sample preparation for gas chromatography. SPME reduces the time required for sample preparation and eliminates the use of large volumes of extraction solvents. An SPME/HPLC interface now enables analysts to use SPME in analyses of weakly volatile or thermally labile compounds, such as food components and pharmaceutical compounds. Among the recently developed applications for SPME/HPLC are procedures for monitoring antioxidants and preservatives in foods and beverages.*

### Key Words:

- antioxidants • preservatives • foods • SPME
- sample preparation

Because analytes are rapidly extracted from a sample matrix, with virtually no solvent consumption, solid phase microextraction (SPME)\* is widely regarded as a practical approach to sample preparation for gas chromatography. SPME saves preparation time and solvent purchase and disposal costs, and can improve the limits of detection. A recently developed SPME/HPLC interface enables analysts to use SPME in analyses of many weakly volatile or thermally labile analytes (1).

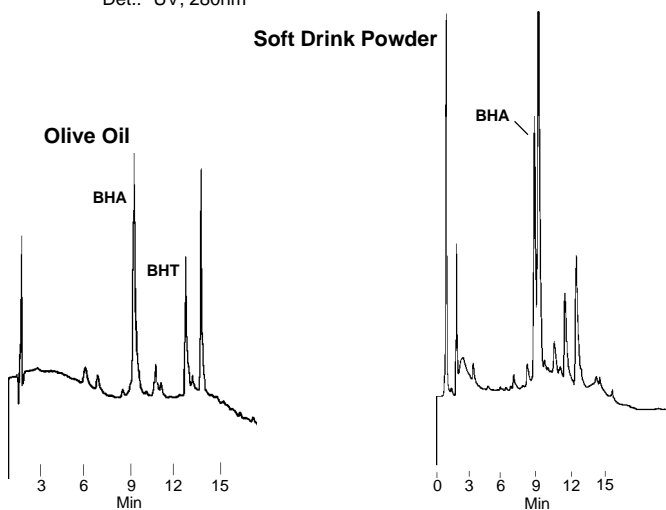
Antioxidants and preservatives maintain the quality of food products during storage. These additives must be monitored to ensure that they are within specified limits. Currently, BHA (2- and 3- tert-butyl-4-hydroxyanisole) and BHT (3,5-di-tert-butyl-4-hydroxytoluene) in oils and fats are analyzed through a process that includes tedious liquid extraction/concentration (2). SPME/HPLC offers a considerable savings of time and solvents, while providing very satisfactory results, as shown by analyses of BHA and BHT extracted from olive oil and soft drink powder (Figure A). Extraction times of 20 minutes or less provide repeatable results. For these less volatile molecules, extraction by immersing the SPME fiber into the sample is more effective than sampling the headspace.

The current procedure for monitoring benzoic acid in foods includes analyte derivatization, as well as liquid-liquid extraction and concentration of the extract (3). SPME/HPLC eliminates all of these steps and is complete in less than 30 minutes (Figure B). Note in Figure B that caffeine, as well as benzoic acid, is extracted by the 40 $\mu$ m polydimethylsiloxane/divinylbenzene SPME fiber.

These examples show SPME now offers a faster, better approach to sample preparation for monitoring food preservatives by HPLC. The combination of SPME with HPLC should prove valuable in many other food and beverage applications, as well as in pharmacological, environmental, and other applications involving molecules unsuited to analysis by gas chromatography.

### Figure A. Rapid Extraction and Analysis of Antioxidants in Foods

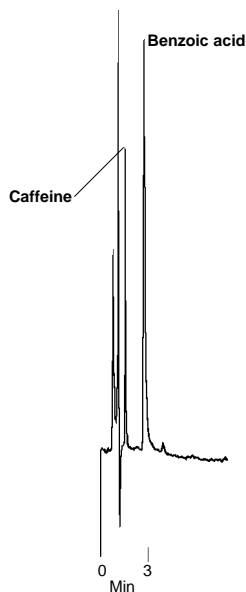
Sample: 3mL olive oil containing 1mg/g each analyte  
0.6g soft drink powder in 3mL water  
SPME Fiber: 40 $\mu$ m PDMS/DVB-HPLC  
Cat. No.: 57317  
Extraction: immersion, 15 min (olive oil) or 20 min (powder), rapid stirring  
Desorption: static, acetonitrile:methanol, 50:50, 5 min (olive oil) or 3 min (powder)  
Column: SUPELCO SIL LC-18, 15cm x 4.6mm ID, 5 $\mu$ m particles  
Cat. No.: 58230-U  
Mobile Phase: A = 5% acetic acid in water  
B = acetonitrile:methanol, 50:50  
30% B for 2 min, then to 100% B over 10 min, hold 10 min  
Flow Rate: 2mL/min  
Det.: UV, 280nm



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## Figure B. Monitor Benzoic Acid in Less Than 30 Minutes, Without Derivatization

Sample: diet cola in water, 1:10 (pH to 2.2 with acetic acid), 3mL  
 SPME Fiber: **40µm PDMS/DVB-HPLC**  
 Cat. No.: **57317**  
 Extraction: immersion, 25 min (rapid stirring)  
 Desorption: static, mobile phase, 1 min  
 Column: **SUPELCOSIL LC-18, 15cm x 4.6mm ID, 3µm particles**  
 Cat. No.: **58985**  
 Mobile Phase: acetonitrile:0.02M sodium acetate (pH 4.3 with acetic acid), 20:80  
 Flow Rate: 1.5mL/min  
 Det.: UV, 254nm



796-0496

### References

- For descriptive information about the HPLC/SPME interface request Product Specification 496049.
  - AOAC Official Method 983.15, *AOAC Official Methods of Analysis*, March 1995 Supplement.
  - AOAC Official Method 983.16, *ibid.*
- References 2 and 3 not available from Supelco. Obtain from AOAC International, Customer Services, 481 N. Frederick Avenue, Suite 500, Gaithersburg, MD 20877-2417 USA

\* Technology licensed exclusively to Supelco. US patent pending; European patent #0523092.

## Ordering Information:

Description	Cat. No.	
SPME Holder for HPLC**	57331	
SPME/HPLC Interface with Valco® Valve	57350-U	
with Rheodyne® Valve	57353	
SPME Fiber Assembly 40µm PDMS/DVB-HPLC	57317	
SUPELCOSIL™ LC-18 HPLC Columns 15cm x 4.6mm ID, 5µm particles	58230-U	
15cm x 4.6mm ID, 3µm particles	58985	
Phenolic Antioxidants Kit Individually packaged neat, under nitrogen, in quantities listed below.	47192	
<b>Component</b>	<b>CAS No.</b>	<b>Qty.(mg)</b>
Propyl gallate (PG)	121-79-9	500
2,4,5-Trihydroxybutyrophenone (THBP)	1421-63-2	500
tert-Butylhydroquinone (TBHQ)	1948-33-0	500
Nordihydroguaiaretic acid (NDGA)	500-38-9	100
2- & 3-tert-Butyl-4-hydroxyanisole (BHA)	25013-16-5	500
2,6-Di-tert-butyl-4-hydroxymethylphenol (Ionox 100)	88-26-6	100
3,5-Di-tert-butyl-4-hydroxytoluene (BHT)	128-37-0	500
Lauryl gallate (Dodecyl gallate)	1166-52-5	500
Octyl gallate	1034-01-1	500
Ethoxyquin***	91-53-2	500

\*\* Initially you must order both holder and fiber assembly. Holder is reusable indefinitely.

Cat. No. 57331 also is used with Varian 8100/8200 AutoSampler (requires Varian SPME upgrade kit, available from Varian).

\*\*\*Qualitative standard. Purity >70% cannot be assured.

For descriptions of our SPME fibers and accessories, request publication 413019.

For individual chemical standards for antioxidants/preservatives, refer to the Supelco catalog.

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