

## Determination of Preservatives in Foodstuffs and Cosmetics by HPLC

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### Introduction:

The inspection of adherence to legal limits of preservatives in foodstuffs and cosmetics can be performed by HPLC. The typical concentrations of preservatives found in directly consumable commercial foodstuffs lie around 0.1 % for sorbic acid and benzoic acid, and around 0.05 % for para-hydroxy benzoic acid (PHB) and PHB-ester. Determination of the preservatives found in such extracts is done through HPLC and subsequent UV detection at various wavelengths. Identification of the substances present is made through their spectrums and retention times. This is of advantage because a variety of the sample's components also absorb at 235 nm, such as artificial sweeteners and antioxidants.

With a Smartline HPLC system from Knauer, an uncomplicated and fast analysis of preservatives can be carried out using a Eurospher 100 C8 column. The Smartline UV Detector 2600 makes it possible to switch across wavelengths and simultaneously acquire spectrums throughout the entire analysis. The Smartline system consists of the Pump 1000, the multi-wavelength UV Detector 2600 with DAD technology, the Autosampler 3800, the Manager 5000 with degasser and low pressure gradient module and the Column Oven 4000.

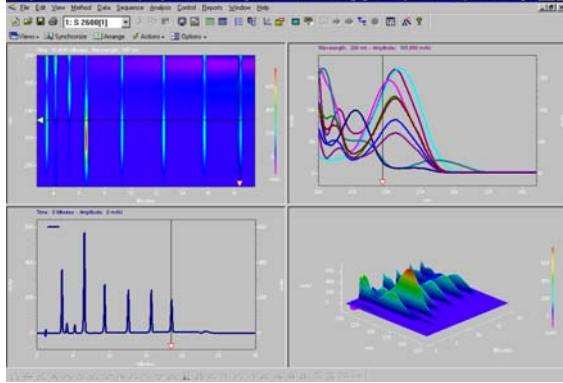
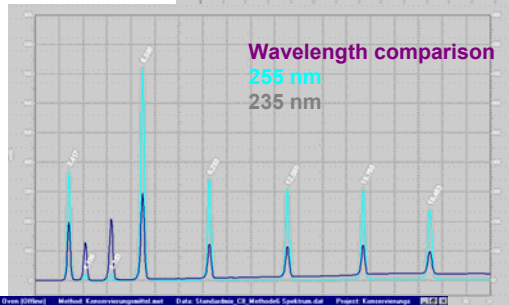
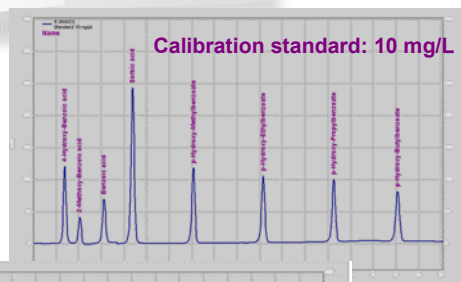
### Method parameters:

Column: Eurospher 100, 125 x 4 mm (B7Y529)  
 Eluent: A: Ammonium formate buffer / methanol 50 / 20  
 B: Ammonium formate buffer / methanol 50 / 70  
 Gradient: 0 – 15 min: 100 % A to 100 % B  
 15 – 20 min: 100 % B  
 Flow rate: 1 ml/min  
 Temperature: 30 °C  
 Injection volume: 20 µl  
 Detection: UV (Smartline 2600) with wavelength switching:  
 0 – 3.75 min: 255 nm  
 3.76 – 5.70 min: 235 nm  
 5.71 – 25 min: 255 nm

Stock solutions of each of the standards (sorbic acid, benzoic acid, PHB, PHB-met, PHB-eth, PHB-prop, PHB-but and 2-methoxybenzoic acid (ISTD)) were prepared in water/methanol (60/40, v/v) at a concentration of 1 mg/ml. The chromatographic conditions (eluent concentration, flow rate, temperature) were optimized in such a way that the preservatives analyzed could be separated as fast as possible in one run. The concentration range for the calibration was in the area of 10 mg/L to 50 mg/L.

### Results:

All of the standards gave very good linear calibration curves with regression coefficients ( $r^2$ ) of 0.9998 or better. With of wavelength switching, a sufficiently detection was possible. The following limits were obtained: PHB = 0.5 mg/L, 2-methoxy benzoic acid = 2.4 mg/L, benzoic acid = 1.4 mg/L, sorbic acid = 0.25 mg/L, PHB-met = 0.52 mg/L, PHB-eth = 0.57 mg/L, PHB-prop = 0.6 mg/L, and PHB-but = 0.75 mg/L. By way of the spectrum check feature, an additional assurance of the sample values obtained was possible. This feature is useful for confirming the identity of each preservative peak, particularly when analyzing foodstuff and cosmetic matrices in which additional components that coelute with the preservatives of interest are common. Such coelution can be simply detected with the peak purity function during spectrum acquisition.



the use sensitive detection