Sensitive online SPE determination of bisphenol A in water samples

An HPLC system with additional switching valves can be used in continuous online SPE operation without time-consuming sample preparation steps

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Abstract:
In this application note a method for the sensitive determination of bisphenol A (BPA) from water samples is presented. Due to the online SPE coupling, no time consuming and manual sample preparation steps are needed. This makes the method well-suited for routine analyses of BPA in low concentrated samples like drinking water.

Body text:

Introduction

Solid-phase extraction (SPE) is an effective preparation method for concentrating analytes prior to HPLC analysis. Classically, this method is done offline via time consuming steps. The advantages of online coupling result in a reduction of analysis time, sample contamination, and analyte loss. This automated method is perfectly suited for pre-concentration of Bisphenol A (BPA) in drinking water. This substance is known for its endocrine effects similar to the hormone estrogen even at very low dosage and is associated with environmental and health problems. Derived from various studies a maximum entry of <1 µg/ml is expected in cold drinking water. In warmed-up water (70°C) a concentration of up to 30 µg/ml is possible. All these facts make it inevitable to establish a fast HPLC method that reaches very low detection limits for BPA.

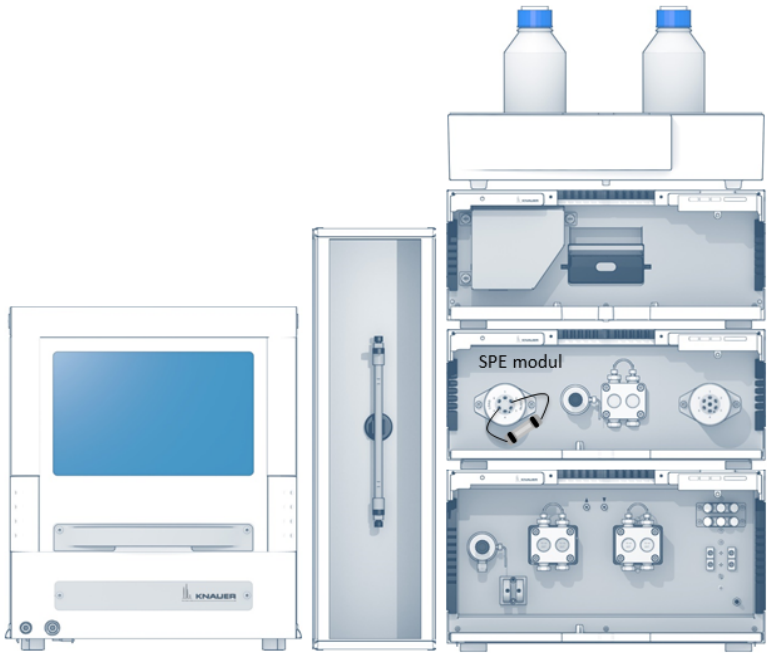

Figure 1: Schematic arrangement of the involved system components for the HPLC system coupled to online SPE extraction

Experimental preparation of standard solution

All standards were prepared and diluted with LC-MS grade water to eliminate matrix effects during calibration and to ensure high quality of the standard.

Analytical HPLC method parameters

Column	Eurospher II 100 C18 A, 3 µm, 100 x 3 mm
Eluent A	Water
Eluent B	Acetonitrile
Gradient	Isocratic 50 % B
Flow rate	0.6 ml/min
Injection volume	10 µl
Column temperature	30 °C
System pressure	approx. 230 bar
Detection	UV at 227 nm
Run time	5 min

SPE method parameters

SPE column	Eurospher II 100 C18 A, 3 µm, 30 x 4 mm	
Step 1	Flush the extraction column with 100 % water	Sample extraction
Step 2	Switch to the sample and extract it for 15 minutes	
Step 3	Flush again with 100 % water	
Step 4	Switch the extraction column into the determination part of the HPLC Plus system	Sample analysis
Step 5	After switching back, flush with 100 % acetonitrile	Cleaning of Extraction column
Step 6	Flush with water until the end of method	

Results

After calibration by direct injection, the recovery rate is determined with the online SPE column in the flow path. Differing concentrations down to 0.07 ng/ml have been extracted from prepared water samples with constant extraction time. Afterwards the extraction time was varied using a solution with a constant concentration of 0.1 ng/ml. A recovery rate of 98% for BPA was found.

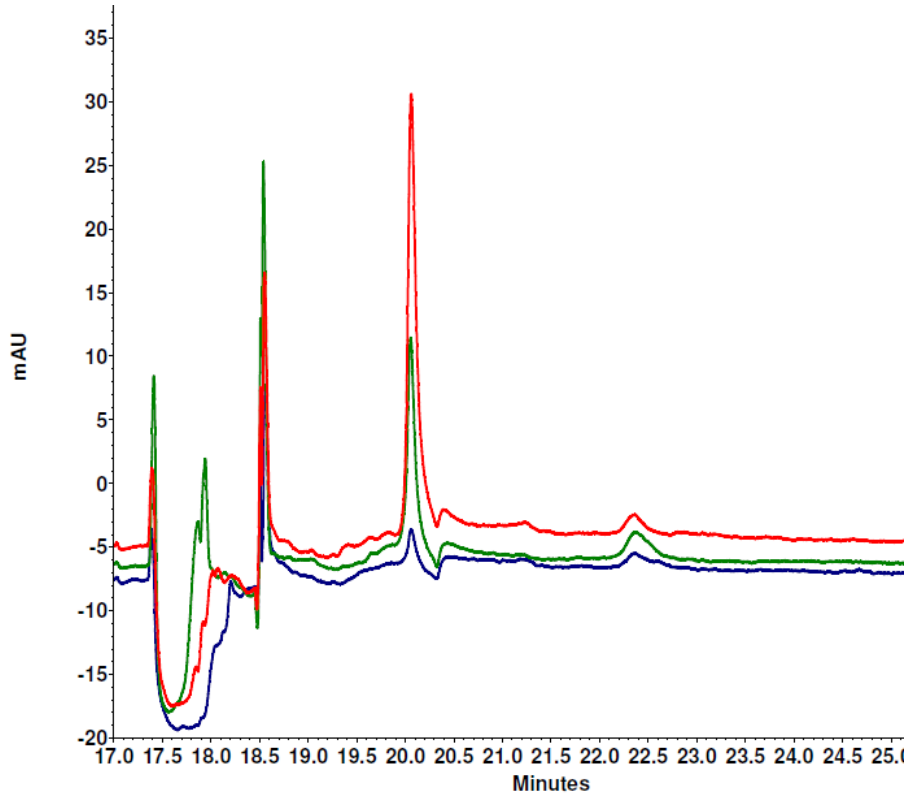

Figure 2: Chromatogram of three different concentrations with the same online SPE extraction time

Conclusion

The presented automated method is well-suited for the sensitive analysis of BPA in water samples like drinking water. For a lower detection limit, the online SPE extraction time can simply be increased. The method makes time consuming manual sample preparation steps obsolete.