Science Together

KNAUER

Sensitive online SPE determination of bisphenol A in water samples

Juliane Böttcher, Kate Monks; applications@knauer.net
KNAUER Wissenschaftliche Geräte GmbH, Hegauer Weg 38, 14163 Berlin; www.knauer.net.

SUMMARY

In this application a method for the sensitive determination of bisphenol A (BPA) from water samples is presented. The use of online solid phase extraction (SPE) coupling avoids time consuming and manual sample preparation steps, making the method well-suited for routine analyses of BPA in low concentration samples like drinking water.

INTRODUCTION

Solid phase extraction 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 BPA in drinking water. The main source for BPA is the industrial production of polycarbonates and polyvinyl chloride (PVC) where it is a major constituent. It is also an important monomer in the production of polycarbonate. BPA is known for its endocrine effects similar to the hormone estrogen even at very low dosage and is associated with environmental and health problems. Based on previous studies a maximum entry <1 µg/ml in cold drinking water is expected. In warmed-up water (70 °C) a concentration up to 30 µg/ml is possible.

Additional Information

Sensitive online SPE determination of bisphenol A in water samples

RESULTS

After calibration by direct injection using an autosampler, 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. **Fig. 1** shows the chromatogram of three different concentrations with same online SPE extraction time. **Fig. 2** shows an original drinking water sample spiked with BPA. 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.

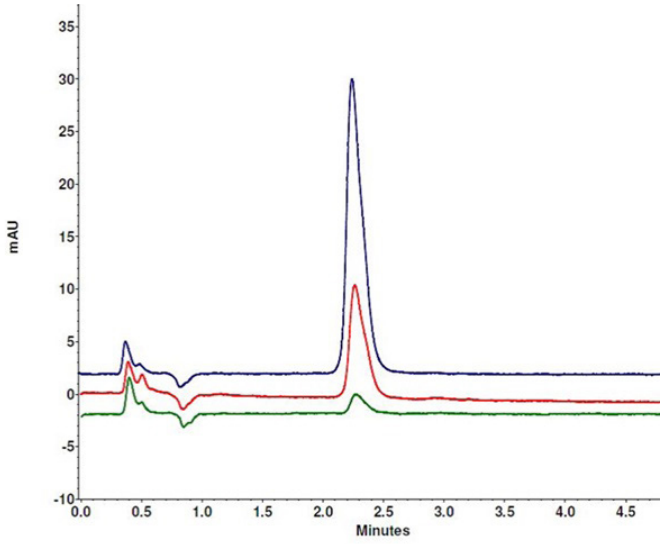

Fig. 1 To determine the recovery rate of calibration points, two different methods are taken as a basis. First three differing concentration (c1=0.07 ng/ml, c2=0.4 ng/ml, c3=1 ng/ml) have been extracted with the same extraction time. In this part recovery rates of 93 % for bisphenol A were found (n=4 for each concentration).

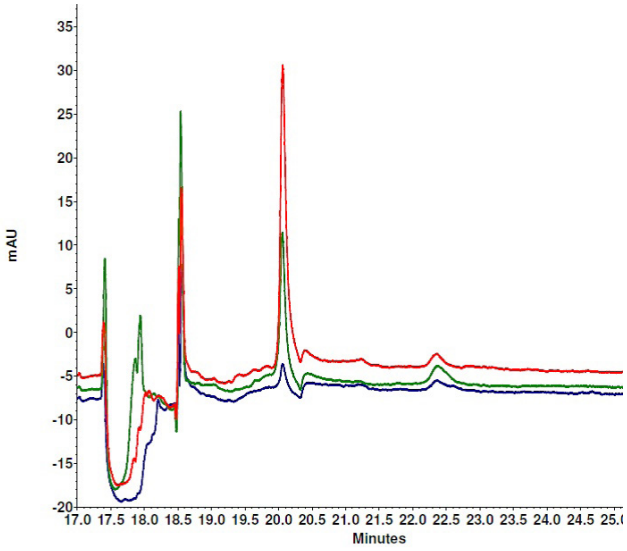

Fig. 2 Chromatogram of three different concentrations with same online SPE extraction time.

SAMPLE PREPARATIONS

An AZURA® Analytical HPLC Plus system was used for this application. It consists of an AZURA P 6.1L LPG pump, an autosampler AZURA AS 6.1L, an AZURA CT 2.1 column thermostat, a AZURA MWD 2.1L multi wavelength detector and an assistant AZURA ASM 2.2L equipped with a 12 port multi position valve, a 6 port/2 position injection valve and a pump with 10 ml pump head. The analytical method runs isocratic at a flow rate of 0.6 ml/min with a mixture of acetonitrile and water 50:50 (v/v). The column thermostat was set to 30 °C and the detector recorded at 227 nm. The used columns are filled with KNAUER Eurospher II 100-3 C18A silica. The SPE method parameters are divided into different steps, including column conditioning, sample extraction, sample analysis, and reconditioning of of the SPE column.

CONCLUSION

The method presented in this application note is well suited for the analysis of bisphenol A in water samples like drinking water and allows varying the extraction time dependent on the expected bisphenol A concentration. For a higher and better evaluable peak signal the time the sample flushes over the extraction cartridge can simply be increased. With this sensitive method it is possible to successfully quantify even low concentrated samples and extracts and equipped with the AZURA ASM 2.2L the system can easily be used in continuous operation.

REFERENCES

[1] Ligang Chen, Hui Wang, Qinglei Zeng, Yang Xu, Lei Sun, Haoyan Xu, Lan ding, "Online Coupling of Solid-Phase Extraction to Liquid Chromatography—A Review", Journal of Chromatographic Science, Vol. 47, September 2009

[2] Jordáková I., Dobiás J., Voldrich M., Poustka J. Determination of Bisphenol A, Bisphenol F, Bisphenol A diglycidyl Ether and Bisphenol F Diglycidyl Ether migrated from food cans using Gas Chromatography- Mass spectrometry. Czech J. Food Sci.2003 Vol. 21, No. 3: 85-90

[3] Umweltbundesamt, Abteilung Umwelthygiene. Telegramm: Umwelt + Gesundheit. „Bisphenol A" – Wir haben ein Problem. Ausgabe 04/2008

[4] Umweltbundesamt, Pressestelle „Bisphenol A Massenchemikalie mit unerwünschten Nebenwirkungen", aktualisierte Fassung Juli 2010, http://www.umweltbundesamt.de/uba-infomedien/mysql_medien.php?anfrage=Kennummer&Suchwort=3782

MATERIALS AND METHODS

Tab. 1 SPE parameters

Step 1 (sample extraction)	Flush the extraction column with 100 % water for 0.5 min at a flow rate of 3 ml/min
Step 2	Switch to the sample and extract it for 15 min (variable) with a flow rate of 3 ml/min
Step 3	Flush again with 100 % water for 1.5 min at a flow rate of 3 ml/min
Step Step 4 (sample analysis)	Switch the extraction column into the determination part of the HPLC system for 3 min, starting the data acquisition immedia-tely after switching
Step 5 (extraction column cleaning)	After switching back, flush with 100 % acetonitrile for 3 min at a flow rate of 3 ml/min
Step 6	Flush with water at a flow rate of 3 ml/min for 5 min and then at a flow rate of 0.5 ml/min until the end of method

Tab. 2 Method parameters

Eluent (A)	Water		
Eluent B	Acetonitrile		
Gradient	Isocratic 50 % B		
Flow rate	0.6 ml/min	System pressure	approx. 230 bar
Column temperature	30 °C	Run time	5 min
Injection volume	10 µl	Injection mode	Full loop
Detection wavelength	227 nm	Data rate	20 Hz
		Time constant	0.05 s

Tab. 3 System configuration

Instrument	Description	Article No.
Pump	AZURA P 6.1L, LPG 10ml, SSt	APH34EA
Autosampler	AZURA AS 6.1L	AAA01AA
Detector	AZURA MWD 2.1L	ADB01
Flow cell	LightGuide 50mm, 6µl	AMD59
Assistant	AZURA ASM 2.2L	AY00562
Thermostat	AZURA CT 2.1 Column Thermostat	ATC00
Eluent tray	AZURA ET 2.1L	AZC00
Column	Vertex Plus Column, 100x3 mm ID, Eurospher II 100-3 C18A	10CE184E2G
Column SPE	Vertex Plus Column, 30x4 mm ID, Eurospher II 100-3 C18A	03DE184E2G
Software	OpenLAB CDS EZChrom Edition	A2600-1