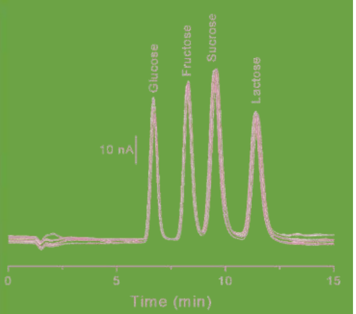
BISPHENOL A IN WATER

THE FINEST LC-EC APPLICATIONS FOR
FOOD & BEVERAGE ANALYSIS
EVER PROCESSED

Bisphenol A
Catechins
Flavonoids and phenols
Phenols
Antioxidants

Polyphenols
Resveratrol
Epicatechin
Quercetin
other polyphenols

Carbohydrates
Iodide
Vitamins A, C, D, E, and K
Q10
ubiquinols



INTRODUCTION

Bisphenol A (BPA) is an important chemical building block that is used primarily to make polycarbonate plastic and epoxy resins, both of which are used in a wide variety of applications. Common examples of polycarbonate products include eyeglass lenses, digital media (e.g., CDs, DVDs), electronic and electrical equipment housings (e.g., personal computers, appliances, power tools), automobile headlight lenses, sports safety equipment (e.g., helmets, goggles), and reusable food and drink containers. When used as a coating on the interior of metal cans, epoxy resins protect the integrity and quality of our food supply by preventing corrosion and contamination of canned foods and beverages with metals.

- Flexcell with echangeable working electrode
- Magic Diamond working electrode
- Robust & reproducible Analysis

Summary

Concerns have been expressed about the estrogenicity of BPA and other aromatic components that leached from commercial products into food and beverages. In this note a sensitive method is presented to analyse drinking water from bottles and cans on the presence of BPA.
An ALEXYS system is used in combination with a solid phase sample pre-concentration step. A detection limit of 0.5 nmole/L has been obtained.

Fig. 1. ALEXYS Bisphenol A Analyzer.

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Method

Table 1	
LC-EC Conditions	
HPLC	ALEXYS Bisphenol A Analyzer
Temp.	35 °C (separation and detection)
Column	Analytical: ALF 215 150 X 2.1 mm, 3µm ALH pre-column 5 x 1 mm, 5µm
Flow cell	VT-03 with 2 mm GC WE, HyREF
Flow rate	A: 200 µL/min B: 800 µl/min (pump 2, pre-concentration)
Pressure	A: 9 MPa
Ecell	900 mV

An ALEXYS system is used in combination with a solid phase sample pre-concentration step. A 1 mL sample volume is loaded onto a pre-column by a user program in the AS 100 autosampler. For this purpose the AS 100 has been equipped with a 10-port valve. A second pump is used to supply the washing solvent. Advantages of using the pre-column is not only an improvement of detection limit, but also the chromatography and sample clean-up. Experiments with 1000 µL injections without pre-column showed a large front peak and a considerable shift in retention times. By applying a pre-column this front peak and also the drift in the baseline are significantly reduced.

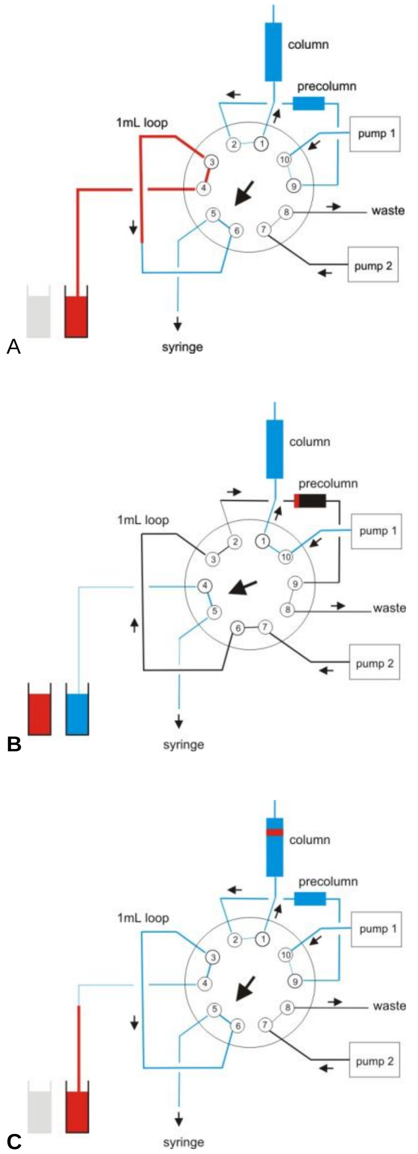

Fig. 3. User program for BPA analysis. A 1 mL loop is filled with sample [A]. The 10-port valve switches and pump 2 with solvent B carries the sample onto the pre-column [B]. The valve switches back and the pump 1 with solvent A flushes the concentrated sample from the pre-column onto the analytical column. Meanwhile, filling of the 1 mL sample loop is started.

Hydrodynamic voltammogram

To find the optimum detection potential BPA was analysed at potential settings between 500 and 1200 mV (vs HyREF) using 10 µL injections of a 500 nmol/L BPA standard. The optimum potential that gives the best s/n ratio is 900 mV.

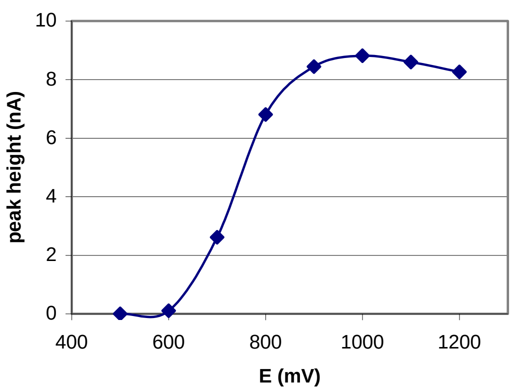

Fig. 2. Hydrodynamic voltammogram for BPA.

For the construction of a hydrodynamic voltammogram a 3mm ID column and a flow cell with 3 mm working electrode has been used.

Electrode contamination

Analysis of relatively high concentrations of BPA showed a decrease in peak height of about 50% after 100 injections. After polishing the working electrode the signal was restored to the original peak height. This shows that at elevated concentrations of

BPA electrode contamination is an issue. At lower concentration levels no contamination has been observed

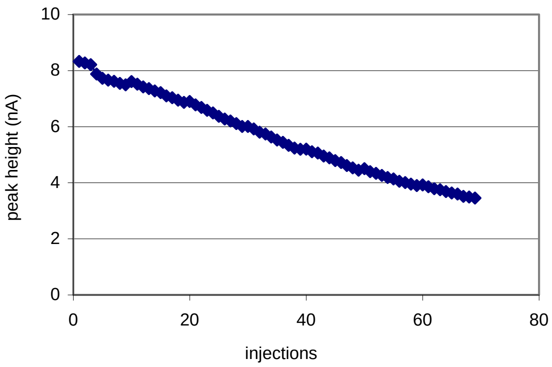

Fig. 4. Decrease in peak heights of 500 nM BPA (10 µL) on a 3 mm column due to electrode contamination.

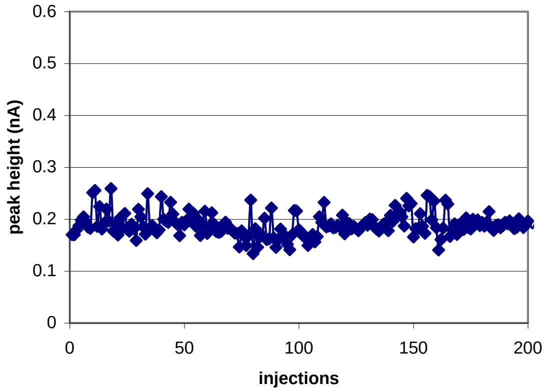

Fig. 5. Peak heights of 5 nM BPA (10 µL) on a 2 mm column, 200 replicate injections. No electrode contamination has been observed.

In cases where electrode contamination is an issue a cleaning pulse can be applied. Typically, the potential is set to a reductive potential for about one minute. After about one minute the detection potential is applied and after a few minutes stabilisation period the system is ready for analysis. We found that switching the potential to +100 mV for 54 s is sufficient to prevent electrode contamination.

Linearity

The linearity has been studied in the range 0.1-10 nM BPA. Below 0.5 nM the results were not linear and these data points are rejected (Fig. 6). Correlation coefficient r is better than 0.999. From this calibration data a detection limit of 0.3 nM was found. Calculation using the signal-to-noise ratio [LOD = 3 n c / s] results in a factor 10 better detection limit. However, practical detection limit is higher because of non-linearity at lower concentrations.

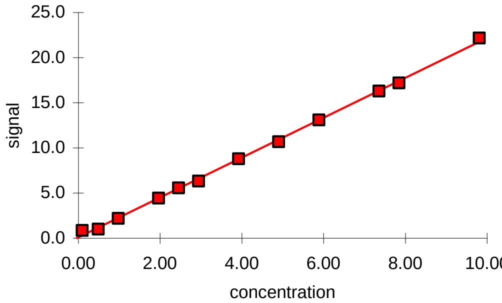

Fig. 6: Calibration curve of BPA: Y = 0.0843 + 2.2072 X. Signal is peak height in nA, concentration in nmol/L.

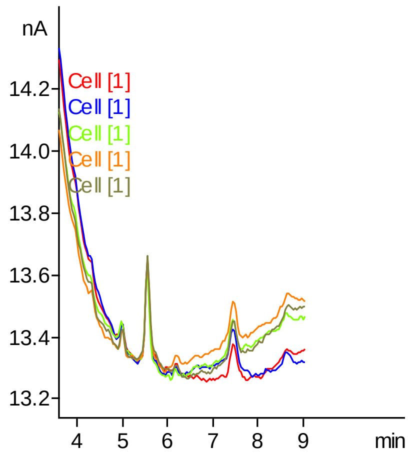

Fig. 7: Overlay chromatograms of reproducibility study of 0.5 nM BPA (5.5 min, n=5)

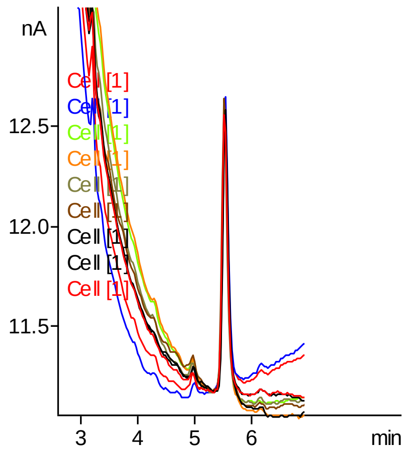

Fig. 8: Overlay of chromatograms of 2.5 nM BPA (n=9)

Reproducibility

Reproducibility is studied for standard injections of 0.5 nM, 1 nM and 2.5 nM BPA (1000 µL). In Fig. 7 - Fig. 8 overlays of chromatograms of 0.5 nM and 2.5 nM BPA are shown. The results are summarized in Table 2. For areas and peak heights the %RSD is the same, about 2%.

Table 2

Repeatability

C (nM)	t (min)	%RSD	h (nA)	%RSD	n
0.5	5.52	0.1	0.33	1.9	5
1.0	5.52	0.1	0.64	1.2	8
2.5	5.51	0.1	1.45	1.8	9

Analysis of drinking water

Drinking water from 2 different PET bottles (Spa, Sourcy) and from a polycarbonate container of an aqua machine (Nestlé) was analysed. The PET bottles were free of BPA. In water from the aqua machine a concentration level of 1.5 nM BPA was found.

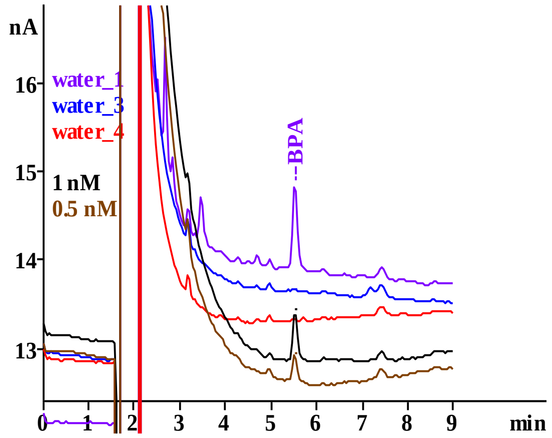

Fig. 9: Overlay of chromatograms of water samples (3 upper traces) and standards (2 lower traces: 0.5 and 1 nmol/L). Only water sample no 1 contained BPA (1.5 nmol/L).

Conclusion

An ALEXYS system has been used for the analysis of BPA in drinking water. A 10-port valve was used in combination with a pre-column for sample pre-concentration and clean-up. A detection limit of 0.3 nM has been obtained. At 0.5 nM an RSD of better than 2% for peak heights and areas was found.

References

1. Environmental Health Perspectives 108 (1) (2000) 21. *Determination of Bisphenol A and Related Aromatic Compounds Released from Bis-GMA-Based Composites and Sealants by High Performance Liquid Chromatography*
2. Journal of Chromatography B, 736 (1999) 255-261. *Sensitive method for the determination of bisphenol-A in serum using two systems of high-performance liquid chromatography*

PART NUMBERS	
180.0090B	ALEXYS Bisphenol A Analyzer
250.1120	ALF-215 column, 150x2.1mm, 3um C18
250.1132	ALH pre-column cartridge 5 x 1 mm ID, 5 µm, 5pk
250.1134	ALH pre-column holder, 5mm