

H.J Brouwer, L. van Heerwaarden, N.J. Reinhoud and M. Eysberg
Antec Leyden, Industrieweg 12, 2382 NV Zoeterwoude, The Netherlands

Introduction

Bisphenol A (BPA) is an important chemical building block and additive in a wide variety of plastics. Its worldwide manufacture is approximately 3.2 million metric tons/year. BPA is the key monomer in production of polycarbonate plastic and epoxy resins and is furthermore used as an antioxidant, plasticizer and as a polymerization inhibitor in PVC. Polycarbonate plastics have many applications including use in food and drink packaging, e.g. water and infant bottles, compact discs and impact-resistant safety equipment. Epoxy resins are used as lacquers to coat metal products such as food cans and water supply pipes. Concerns about the estrogenicity of BPA leaching from commercial products has been expressed [1]. The free monomers can be detected by different analytic methods [2, 3].

A sensitive LC-EC method is presented to analyse drinking water from bottles and cans on the presence of BPA. An ALEXYS LC system with electrochemical detection in combination with a solid phase sample pre-concentration step has been applied.



Fig. 1. ALEXYS[®] LC-EC system for the analysis of Bisphenol A with a second pump for pre-concentration of sample.

Method

HPLC ALEXYS Bisphenol A analyzer (p/n 180.0090A)
Flow cell VT-03 with 2 mm GC WE, HyREF™
Ecell 900 mV
Temperature 35 °C (separation & detection)
Flow rate A: 200 µL/min (separation) , B: 800 µL/min (pre-concentration)

For sample clean-up and improved detection limits for BPA an automated solid phase sample pre-concentration step is applied using the AS 100 autosampler configured with a 10 port injection valve. A 1 mL sample volume is loaded onto a pre-column by a user program in the AS 100 autosampler.

Electrode contamination

Analysis of relatively high concentrations of BPA showed a decrease in peak height of about 50% after 100 injections. At lower concentration levels no 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. We found that switching the potential to +100 mV for 54 s is sufficient to prevent electrode contamination.

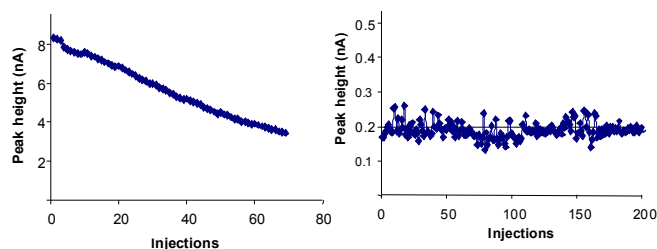


Fig. 2. Left side: peak heights of 70 replicate injections of 500 nM BPA (10 µL). A decrease of peak height is observed due to electrode contamination. Right side: peak heights of 200 replicate injections of 5 nM BPA (10 µL). No electrode contamination has been observed.

Reproducibility, Linearity & LOD

The reproducibility was studied for low concentrations of BPA standards of 0.5 nM, 1 nM and 2.5 nM (1 mL injection volume). The linearity has been studied in the range 0.5-10 nM (correlation coefficient r better than 0.999). From this calibration data a detection limit of 0.3 nM was found.

Table I. Results of reproducibility study.

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

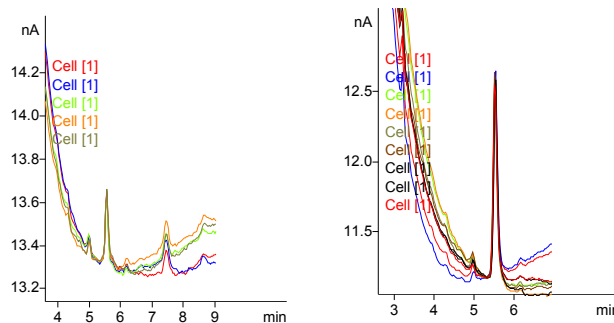


Fig. 3. Left side: Overlay of chromatograms of 2.5 nM BPA (n=9). Right side: Overlay chromatograms of reproducibility study of 0.5 nM BPA (5.5 min, n=5).

Analysis of Drinking Water

Drinking water from 2 different PET bottles and from a polycarbonate container of an aqua machine 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.

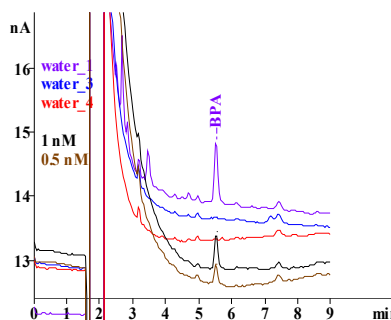


Fig. 4. Overlay of chromatograms of water samples (3 upper traces) and standards (2 lower traces: 0.5 and 1 nmol/L). Only water sample no 1 contained BPA (1.5 nmol/L).

Conclusion

The ALEXYS Bisphenol A analyzer is a dedicated LC system for the trace analysis of BPA in drinking water. The system combines excellent performance with ease of use. The method includes a fully automated sample pre-concentration step and shows good linearity, reproducibility and sensitivity. Concentrations as low as 0.3 nM BPA can be detected.

References

1. F.S. vom Saal, C. Hughes, *Environ. Health Perspect.* **113**(8), (2005) 926–33
2. R Pulgar et al., *Environmental Health Perspectives*, **108**(1), (2000), 21
3. J. Sajiki et al., *Journal of Chromatography B*, **736**, (1999) 255-261