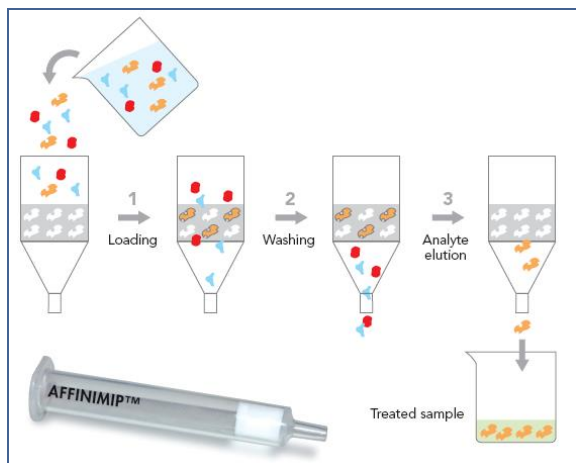


Selective Solid Phase Extraction of Bisphenol A from Beer at Low Concentrations Using Molecularly Imprinted Polymers



Introduction

Bisphenol A (or BPA) (see Figure 1) is a molecule widely used in industry for the synthesis of polycarbonate plastics and epoxy resins. Polycarbonate plastics are used to make a variety of common products including baby and water bottles. Epoxy resins are used as coatings on the inside of almost all food and beverage cans.

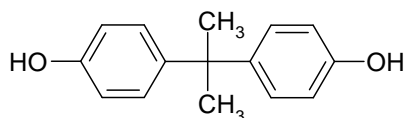


Figure 1. Chemical structure of Bisphenol A

BPA is also an endocrine disruptor, which can mimic the body's own hormones and may lead to negative health effects. The migration of BPA from the packaging to food is the main source of consumers' exposure to BPA. So, the European commission has defined a specific migration limit at a maximum level of 0.6 mg of BPA/kg of food (Directive 2011/8/EU of 28 January 2011). In addition, the directive prohibits the use of BPA to manufacture infant feeding bottles.

BPA is a topical issue with a worldwide regulation going to still lower concentrations of BPA allowed in food. So, highly sensitive and reliable detection methods are required for routine analysis of BPA in food samples.

For such concentrations, a clean-up step is crucial in order to improve the sensitivity, the reliability and the specificity before LC analysis.

To do so, we have developed a new class of intelligent

polymers based on molecularly imprinted polymers specific to Bisphenol A. Molecularly Imprinted Polymer (MIP) is a synthetic material with artificially generated three-dimensional network able to specifically rebind a target molecule. MIP has the advantage to be not only highly selective and specific but also chemically and thermally stable, compatible with all solvents and cost-effective. This polymer is used as a powerful technique for clean-up and pre-concentration applications of Bisphenol A.

This study describes the solid phase extraction of Bisphenol A from Beer using a Molecularly Imprinted Polymer SPE cartridge: **AFFINIMIP® SPE Bisphenols**.

Experimental conditions

Materials

All reagents and chemicals were ACS grade quality or better. Bisphenol A was obtained from Alfa Aesar. Canned Beer and Beer in glass bottle were purchased at a supermarket.

Preparation of samples prior to SPE with AFFINIMIP® SPE Bisphenols Cartridge

The beer is degassed by sonication for 1 hour.

Solid phase extraction (SPE) protocol

The SPE procedure used a 3mL **AFFINIMIP® SPE Bisphenols** cartridge. The details of each step are as follow:

- Condition the SPE cartridge with 5mL of Methanol-2% acetic acid, 5mL Acetonitrile (ACN), then with 5mL of deionized Water
- Load 10mL of degassed beer
- Wash the cartridge with 10mL of deionized Water
- Wash the cartridge with 6mL of deionized Water /Acetonitrile (60/40, v/v)
- Dry 30 seconds
- Elute Bisphenol A with 3mL of Methanol

The SPE procedure lasted approximately 50 minutes.

The elution fraction was then evaporated and dissolved in the mobile phase.

Analysis

HPLC was performed on a ThermoFinnigan Spectra System with a Thermo Hypersil Gold C18 column (150mm x 4.6mm). Separation was carried out using a gradient at a flow rate of 1mL/min. The detection system was a Jasco Model FP-2020 Fluorescence detector set to excitation/emission wavelengths of 230 and 315nm, respectively. The injection volume was 50µL.

Mobile Phase	Time (min)	% Water	% ACN
	0	65	35
	2	65	35
	12	50	50
	12.5	65	35
	22	65	35

Results

Table 1. Recovery of Bisphenol A in beer spiked at different concentrations after AFFINIMIP® SPE Bisphenols clean-up and relative standard deviation calculated from results generated under **repeatability conditions** (n=3).

C° (µg/L)	Mean µg/L	Recoveries %	% RSD _r
1.0	1.1	106.9	1.0
2.0	1.9	93.4	1.0

Table 2. Recovery of Bisphenol A in beer spiked at different concentrations after AFFINIMIP® SPE Bisphenols clean-up and relative standard deviation calculated from results generated under **reproducibility conditions** (n=9).

C° (µg/L)	Mean µg/L	Recoveries %	% RSD _R
1.0	1.0	99.3	8.9
2.0	1.8	90.6	6.0

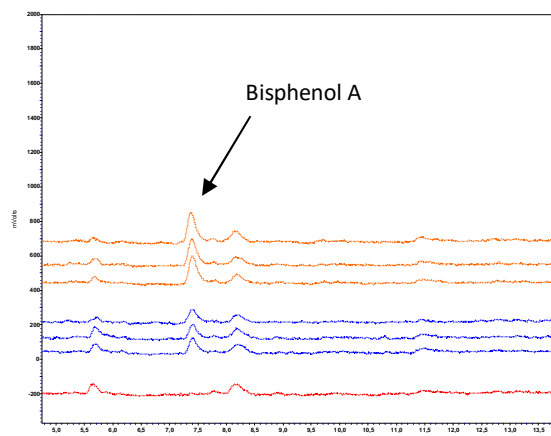


Figure 2. Chromatograms obtained after AFFINIMIP® SPE Bisphenols Clean-up of 10mL of beer spiked at 2µg/L (tested 3 times, orange) or at 1µg/L (tested 3 times, blue) with Bisphenol A or not spiked (red)

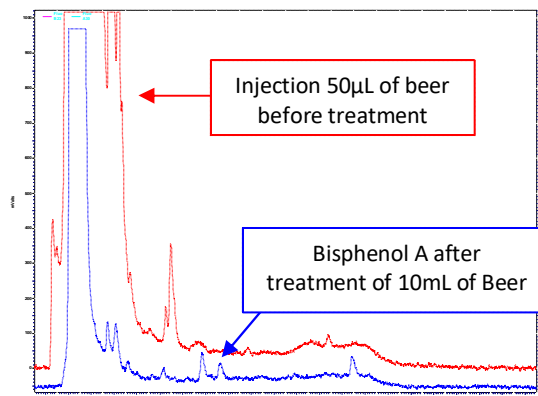


Figure 3. Chromatograms of beer containing 1µg/L of Bisphenol A before (Red) and after (Blue) AFFINIMIP® SPE Bisphenols Clean-up.

Conclusion

AFFINIMIP® SPE Bisphenols cartridge is a simple, fast, sensitive and selective tool for the extraction of Bisphenol A from beer samples with a high recovery yield (> 90%), a good repeatability and reproducibility.

Thanks to the efficient clean-up and a pre-concentration process, a reliable quantification of Bisphenol A at low concentration (1 and 2µg/L) using fluorescence detector is possible. Therefore, **the use of AFFINIMIP® SPE Bisphenols enables to eliminate the tedious derivatization step** required by gas chromatography.

This method is also perfectly suitable for clean-up before GC-MS/MS or LC-MS/MS.

Product reference

- **AFFINIMIP® SPE Bisphenols**

Catalog number: FS106-02 for 25 cartridges
 FS106-02G for 25 Glass cartridges
 FS106-03 for 50 cartridges
 FS106-03G for 50 Glass cartridges