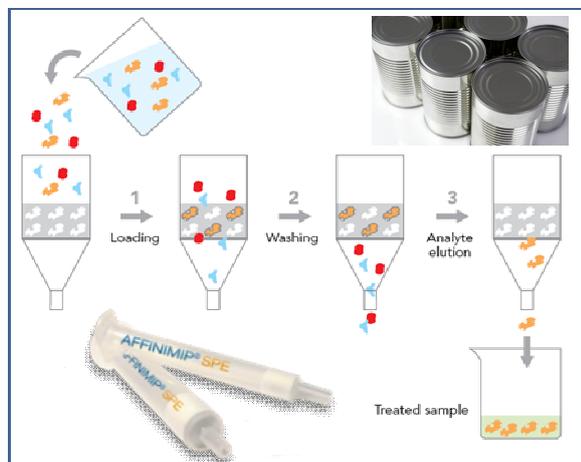


Selective Solid Phase Extraction for Bisphenol A Analysis from Canned Foods At Low Concentrations using AFFINIMIP® SPE Bisphenols



Bisphenol A in food: a routine exposure for consumers that needs to be monitored

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.

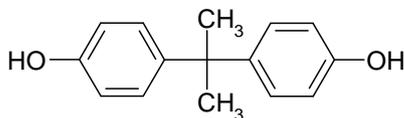


Figure 1. Chemical structure of Bisphenol A

Bisphenol A 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, sensitive and reliable detection methods are required for routine analysis of BPA in food samples.

A clean-up step is crucial in order to improve the sensitivity, the reliability and the specificity before analysis and to remove interfering compounds.

How to solve this?

AFFINISEP has developed a new class of intelligent polymers based on molecularly imprinted polymers (AFFINIMIP®) specific to Bisphenol A used as a powerful technique for clean-up and pre-concentration.

AFFINIMIP® SPE Bisphenols cartridge is a simple, fast, sensitive and selective tool for the extraction of Bisphenol A from complex matrices such as canned foods.

We demonstrate in this application note that a reliable quantification of Bisphenol A from canned foods at low concentrations (1µg/L) using AFFINIMIP® SPE Bisphenols and a fluorescence detector is possible.

In addition, 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.

Results

Clean extracts at Low concentrations: Tests in canned peas and carrots

With a fluorescence detector, we managed to quantify Bisphenol A from canned peas and carrots at 1µg/L with recoveries higher than 95% and a reproducibility relative standard deviation RSD_R of 5%. Lower levels of quantification are expected with LC-MS/MS or GC-MS/MS.

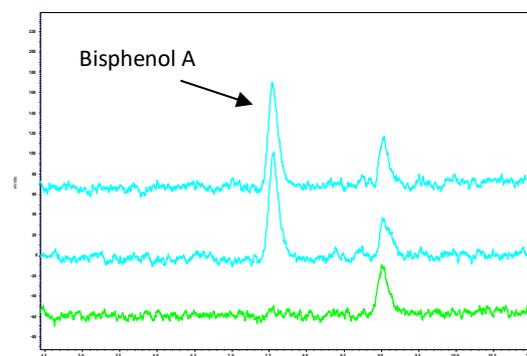


Figure 2. Chromatograms obtained after clean-up with AFFINIMIP® SPE Bisphenols of 10mL of canned Peas and carrots under liquid form spiked with Bisphenol A at 1µg/L (tested twice, blue) or not spiked (green).

High analyte recovery and reproducibility

C° (µg/L)	Mean (µg/L)	Recoveries %	% RSD _R
1.0	1.05	105.1	5

Table 1. Recovery of Bisphenol A after AFFINIMIP® SPE Bisphenols clean-up of 10mL of canned peas and carrots (liquid) spiked at 1µg/L and relative standard deviation calculated from results generated under **reproducibility conditions** (n=4).

Canned salmon and canned tuna samples were contaminated with Bisphenol A

We decided to evaluate the presence of Bisphenol A in commercially available canned foods: 3 different brands of canned salmon and one of canned tuna were tested.

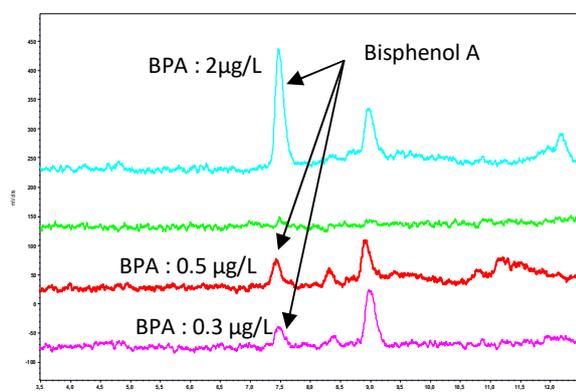


Figure 3. Chromatograms obtained after clean-up with AFFINIMIP® SPE Bisphenols of 10mL of canned salmon and tuna (liquid form).
Blue: 1st price canned salmon containing 2µg/L of BPA
Green: middle grade canned salmon: no BPA was detected.
Red: premium canned salmon containing 0.5µg/L of BPA.
Pink: canned tuna containing 0.3µg/L of BPA.

The results show the presence of Bisphenol A in most samples tested except for middle grade canned salmon. So this last sample was used to validate the clean-up method on these complex matrices.

The method is perfectly suited for all type of canned foods

Similarly to canned peas and carrots, the tests performed in canned salmon demonstrate a high analyte recovery, good reproducibility and clean extracts at low concentrations.

C° (µg/L)	Mean (µg/L)	Recoveries %	% RSD _R
1.0	1.04	104.3	10

Table 2. Recovery of Bisphenol A after AFFINIMIP® SPE Bisphenols clean-up of 10mL of canned salmon (liquid) spiked at 1µg/L and relative standard deviation calculated from results generated under **reproducibility conditions** (n=4).

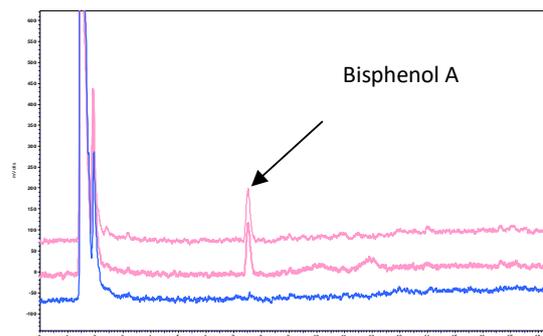


Figure 4. Chromatograms obtained after clean-up with AFFINIMIP® SPE Bisphenols of 10mL of middle grade canned salmon (liquid) spiked with BPA at 1µg/L (tested twice, pink) or not spiked (blue).

Experimental conditions

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 up to 10mL of liquid from canned food (after a filtration using a filter paper (4-7µm))
- 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 FP-2020 with 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
	20	50	50
	20.5	65	35
	35	65	35

Product references:

FS106-02 for 25 cartridges / -02G for 25 Glass cartridges
 FS106-03 for 50 cartridges / -03G for 50 Glass cartridges