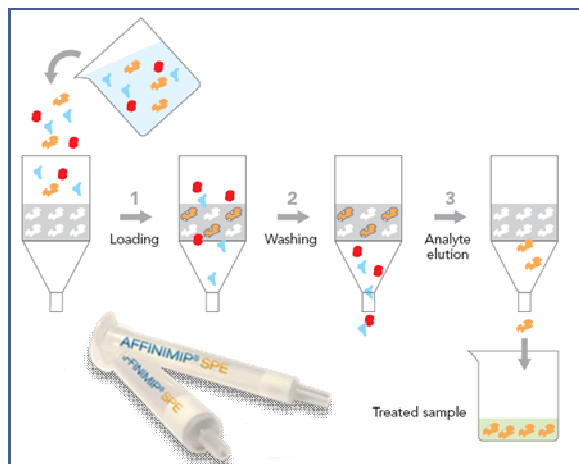
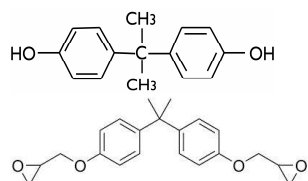


Selective Solid Phase Extraction of Bisphenol A and BADGE from milk in Plastic Food Packaging at Low Concentrations using AFFINIMIP® SPE



Background

Bisphenol A (BPA) and Bisphenol A Diglycidyl Ether (BADGE) are molecules widely used in industry for the synthesis of common products including baby and water bottles and coatings on the inside of food and beverage cans. 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. BADGE is also potential migrant into the preserved food and is of toxicological concern. So, the European commission has defined a specific migration limit at a maximum level of 0.6 mg of BPA/kg of food and has proposed that a limit of restriction of 1 mg/kg food shall include BADGE and its derivatives (Directive 2011/8/EU of 28 January 2011). In addition, the directive prohibits the use of BPA to manufacture infant feeding bottles. Progressively, countries become more and more restrictive on BPA use for food packaging.



Structures of Bisphenol A (BPA) and Bisphenol A Diglycidyl Ether (BADGE)

So, BPA and closely related products are topical issues with a worldwide regulation going to still lower concentrations allowed in food. So, highly sensitive and reliable detection methods are required for routine analysis of BPA and these compounds in food samples, particularly for baby food.

We describe protocols enabling the determination of very low concentration of BPA and BADGE in milk contained in plastic food packaging using AFFINIMIP® SPE Bisphenols cartridge.

We demonstrate in this application note that a reliable quantification of Bisphenol A and BADGE at low concentrations (10µg/kg) 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.

Results

High analyte recovery in milk

Matrice Spiked at 10µg/kg	Mean concentration (µg/kg)	Recoveries %
BPA	10.85	108.5
BADGE	7.5	75

Table 1. Recovery of Bisphenol A and BADGE spiked at 10µg/kg after AFFINIMIP® SPE Bisphenols clean-up of 9mL of milk.

Clean-up at very low concentrations

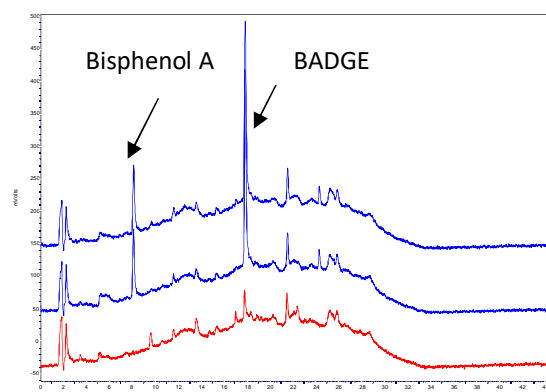


Figure 1. Fluorescence chromatograms obtained after clean-up with AFFINIMIP® SPE Bisphenols of 9mL of milk spiked with 10µg/kg Bisphenol A and 10µg/kg BADGE (tested twice, blue) or not spiked (red).

Experimental conditions

Materials

All reagents and chemicals were ACS grade quality or better. Bisphenol A was obtained from Alfa Aesar and BADGE from Sigma Aldrich. Milk were purchased at a supermarket.

Solid phase extraction (SPE) protocol

The SPE procedure used a 3mL or a 6mL **AFFINIMIP® SPE Bisphenols** cartridge. The details of each step are as follow:

- Condition the SPE cartridge with 3mL of Methanol-2% Formic Acid, 3mL Acetonitrile (ACN), then with 3mL of Water
- Load up to 9mL of Milk
- Wash the cartridge with 9mL of Water
- Wash the cartridge with 6mL of Water/ACN (60/40, v/v)
- Dry 3 minute
- Elute (1) Bisphenol A and BADGE with 3mL of Methanol
- Elute (2) of remaining BADGE with 3mL of Acetonitrile

The elution fractions were gathered and then evaporated and dissolved in the mobile phase.

For the extraction of BADGE, elutions 1 and 2 are required.

Analysis

HPLC was performed on a ThermoFinnigan Spectra System with a Thermo Hypersil Gold 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.

	Time (min)	% Water	% ACN
Mobile Phase	0	65	35
	2	65	35
	12	50	50
	20	20	80
	25	20	80
	30	65	35
	45	65	35

Product references

- **AFFINIMIP® SPE Bisphenols**

- FS106-02 for 25 polypropylene cartridges 3mL
- FS106-02B for 25 polypropylene cartridges 6mL
- FS106-02G for 25 glass cartridges 6mL
- FS106-03 for 50 polypropylene cartridges 3mL
- FS106-03B for 50 polypropylene cartridges 6mL
- FS106-03G for 50 glass cartridges 6mL

Related matrices: canned foods, infant milk, beer, water, urine...