AFFINISEP

APPLICATION NOTEBOOK FOR SAMPLING & SAMPLE PREPARATION

Be selective


Food / Feed QC


Environment


Cosmetics


Pharmaceutical R&D

INTRODUCTION

AFFINISEP offers a comprehensive range of sorbents for the challenging fields of sample preparation, sample clean-up and extraction, from conventional to more sophisticated sorbents. So, for very specific and challenging applications, AFFINISEP has developed AFFINIMIP®SPE products, SPE cartridges based on Molecularly Imprinted Polymers (MIP) which require ready-to-use protocols. AFFINISEP has also developed AttractSPE™ products, SPE cartridges based on classical polymeric sorbents.

In addition, our SPE products experience is continuously enriched with customer interactions and an endless analytical development for new applications. This experience is communicated through Application notes (available on website and via newsletters).

For your convenience, this application notebook will be permanently updated with new protocols and results. Please regularly visit our website **www.affinisep.com** for the latest version of the Application Notebook.
Moreover, we have as well evaluated our products through interlaboratories proficiency testing such as FAPAS and BIPEA. For more information, please contact us at **contact@affinisep.com**.

This Application notebook will be an essential tool to address your technical issues.

TECHNICAL SUPPORT

AFFINISEP has fully integrated technologies platform with specialized teams in organic chemistry, polymer chemistry, analytical and bioanalytical chemistry who are at your disposal to help you in your challenges.

At AFFINISEP, we are committed to providing the best technical support possible. Our Technical Support Group is a team of highly qualified M.Sc. and PhD Chemists, who are at your disposal to resolve your problem and to answer to your queries. For technical inquiries, feel free to contact us either by email: **tech.support@affinisep.com**

We are also very thankful to customer's feedback about our products, protocols and customer services by email to: **contact@affinisep.com**

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	SPE product- ANALYTES	SPE product- ANALYTES
Mycotoxins	Single Mycotoxin	
	Patulin	AFFINIMIP® SPE Patulin
	Zearalenone	AFFINIMIP® SPE Zearalenone
	Ochratoxin A	AFFINIMIP® SPE Ochratoxin A
	Deoxynivalenol (DON)	AFFINIMIP® SPE Deoxynivalenol
	Multimycotoxins	
	Aflatoxins, Ochratoxin A, HT-2, T-2, Fumonisins, Zearalenone, Deoxynivalenol	AFFINIMIP® SPE Multimyco LCMSMS
	Fumonisins AND Zearalenone	AFFINIMIP® SPE FumoZON
Endocrine Disruptor	Estrone, 17α-Estradiol, 17β-Estradiol, Estriol, 17α- Ethynilestradiol	AFFINIMIP® SPE Estrogens
	Bisphenol A, Bisphenol AP, Bisphenol AF, Bisphenol B, Bisphenol S, Bisphenol F…	AFFINIMIP® SPE Bisphenols
	Parabens	AFFINIMIP® SPE Phenolics
	Phenolic compounds	AFFINIMIP® SPE Phenolics
Drug Residues	Amphetamine, Methamphetamine, MDA, MDMA, MDEA	AFFINIMIP® SPE Amphetamines
	Zeranol, Zearalanone, α and β Zearalanol, α and β Zearalenol, Resorcylic acid lactones	AFFINIMIP® SPE Zeranol Residues
	Chloramphenicol	AFFINIMIP® SPE Chloramphenicol
	Tamoxifen	AFFINIMIP® SPE Tamoxifen

See our application notebook for more applications and details…

	SPE product- ANALYTES	SPE product- ANALYTES
Antibiotics and Drugs residues	Nicotine, Procaïnamide	AttractSPE™ HLB
	Caffeine	AttractSPE™ HLB
	Propranolol	AttractSPE™ HLB
	Tetracyclines - Tetracycline, Oxytetracycline, Chlortetacycline, Doxycycline	AFFINIMIP® SPE Tetracyclines
	Sulfonamides – Sulfadimethoxine , Sulfaethoxypyridazine…	AttractSPE™ SCX
	Caffeine, Acetaminophen, Diclofenac, Ibuprofen, Ketoprofen, Naproxen, Carbamazepine	AttractSPE™ HLB
	Antibacterial Aminoglycosides - - Streptomycin, Dihydrostreptomycin,…	AttractSPE™ HLB
	Antibiotics – Quinolones, Macrolides, Lincosamides, Sulfonamides, Penicillins, Cephalosporine, Pleuromutilins, Diamino pyrimidine derivatives	AttractSPE™ HLB
	NSAID (Non Steroidal Anti inflammatory drug) - Salicylic acid, Phenylbutazone, Flunixin, Tolfenamic acid, Meloxicam, Desoximethasome (IS), Ketoprofen	AttractSPE™ HLB
	Penicillin based antibacterials - Ampicillin, Amoxicillin…	AttractSPE™ HLB
	Glucocorticoids - Cortisone, Corticosterone, Aldosterone, Betamethasone, Dexamethasone, Flumethasone, Prednisone, Prednisolone, Methylprednisolone	AttractSPE™ HLB
	Erythromycin and Clindamycin	AttractSPE™ HLB
	Praziquantel and Tiamulin	AttractSPE™ HLB
	Cephalexin	AttractSPE™ HLB
	Quinoxaline-2 -carboxylic acid and 3-methyl quinoxaline-2-carboxylic acid	AttractSPE™ SAX
	Vancomycin	AttractSPE™ SCX
	Valnemulin and Tiamulin	AttractSPE™ HLB
	Phenolic compounds	AFFINIMIP® SPE Phenolics

See our application notebook for more applications and details…

	SPE product- ANALYTES	SPE product- ANALYTES
Pesticides - Herbicides	Glyphosate, AMPA	AFFINIMIP® SPE Glyphosate – AMPA
	Aminopyralid, Clopyralid, Picloram	AFFINIMIP® SPE Picolinic Herbicides
	16 common pesticides - Linuron, Iprodione, Desysopropylatrazine, Desethylatrazine, Aldocarb, Simazine, Carbofuran, Metalaxyl, Atrazin, 2, 4-D, Metazachlor, Dicloran, Phenmedipham, Procymidone, Fenitrothion, Vinclozolin	AttractSPE™ HLB
	Triazine Herbicides - Simazine, Cyanazine, Atrazine…	AttractSPE™ HLB
	Acetamide Herbicides - Metolachlor and metabolites, Alachlor…	AttractSPE™ HLB
	Fungicides - Carbendazim, Thiabendazole	AttractSPE™ SCX
	Pesticides by GC-MS : Metamidophos, Dichlorvos, Acephate, Trifluralin, Diazinon, Chlorothalonil, Dimethipin, Vinclozoline, Methyl parathion, Methyl primophos, Triadimenol-1, DDE, Cypermethrin-3, Difenoconazole-1, Imibenconazole, Tebuthiuron, Bromacil…	AttractSPE™ Carbon/PSA
PAHs	**Hydroxylated Polycyclic Aromatic Hydrocarbons** - 2-Naphtol, 2-Hydroxyfluorene, 9-Phenanthrol…	AFFINIMIP® SPE Phenolics
	Polycyclic Aromatic Hydrocarbons (PAH)	AFFINIMIP® SPE PAH
		AttractSPE™ HLB
		SilactSPE™ CN/SiOH
Phenolics	Guaïacol	AFFINIMIP® SPE Phenolics
	Carnosic acid	AFFINIMIP® SPE Phenolics
	Hydroquinone	AFFINIMIP® SPE Phenolics

See our application notebook for more applications and details…

	SPE product- ANALYTES	SPE product- ANALYTES
Removal of IONS	**Transitions metals ions**	**AttractSPE™ IDA**
	Removal of anionic contaminants and neutralization of highly acidic samples	**AttractSPE™ SAX-HCO3**
	Removal of alkaline earth and neutralization of basic samples	**AttractSPE™ PS-H**
	Removal of Halides ions (chloride, iodide, bromide)	**AttractSPE™ PS-Ag**
	Removal of sulfate ions	**AttractSPE™ PS-Ba**
Biological application	Removal of phospholipids	**AttractSPE™ LipRem**
	Removal of precipitated proteins	**SilactSPE™ Double fritted** & **Single fritted**
	Supported liquid extraction	**SilactSPE™ SLE**
	NNAL	**AFFINIMIP® SPE NNAL**
	Dopamine, Noradrenaline, Adrenaline, ...	**AFFINIMIP® SPE Catecholamines**
	Metanephrine, Normetanephrine and 3-Methoxytyramine, ...	**AFFINIMIP® SPE Metanephrines**
Miscellaneous	Melamine	**AttractSPE™ SCX**
	Cyanuric acid	**AttractSPE™ SAX**
	ARTIFICIAL SWEETENERS - Acesulfame, Aspartame, Cyclamate, Neohespiridine dihydrochalcone, Saccharin, Sucralose	**AttractSPE™ HLB**
	COCAINE AND MAIN METABOLITES - Cocaine, benzoylecgonine and ecgonine methyl ester	**AttractSPE™ HLB**

See our application notebook for more applications and details…

AVAILABLE FORMATS

Open Cartridge	Reversible Cartridge
Format: 1 mL; 3 mL; 6 mL; 15 mL; 20mL; 60mL… **Material:** Polypropylene ; glass (6mL) **Frits:** Polyethylene ; PTFE (glass cartridges); Glass fiber (Glass cartridges)  *Luer compatible*	*Luer compatible* **Format:** 0,7mL ; 2 mL **Material:** Polypropylene **Frits:** Polyethylene  *Luer compatible*
LRC Cartridge	**Cartridge for automate**
Format: 10mL **Material:** Polypropylene **Frits:** Polyethylene  *Luer compatible*	**Format:** 1 mL; 3 mL; 6 mL **Material:** Polypropylene **Frits:** Polyethylene Cartridge for Multipurpose Sampler (GERSTEL) & for ASPEC (GILSON) 
On-line SPE cartridge	**96 Well-plates**
I.D. 2,1 and 4,6mm **Length: 20mm** 	
POCIS	**QuEChERS & Extraction salts**
I.D. 55mm **O.D: 90mm** 	

For each sorbent, the catalog gives references for the most usual formats

If you wish other formats, please contact us

Analysis of MYCOTOXINS

ZEARALENONE IN MAIZE AND RICE

Regulations for unprocessed cereal except maize:
Europe (EC 1126/2007) : 100µg/Kg
Regulations for maize:
Europe (EC 1126/2007) : 350µg/Kg

PROTOCOL OF CLEANUP

Sample preparation

Cleanup with a 3mL/100mg AFFINIMIP® SPE Zearalenone cartridge

25g of ground cereal-based samples were extracted with 100 mL of acetonitrile/deionized water (75/25, v/v) for 3 min. The extract was filtered through a folded filter paper and 10 mL of the filtrate were diluted with 10 mL of deionized water. Then, this solution was filtered through a filter paper.
This solution was used as the loading solution.

Equilibration
3mL Acetonitrile
3mL Water

Loading
12mL of loading solution (eq. 1.5g sample)

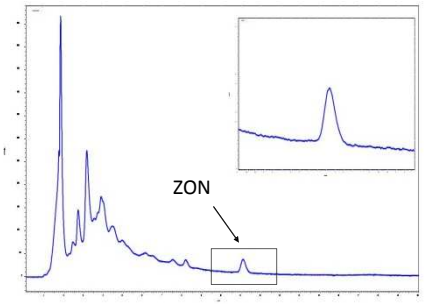
Washing of interferences (W1)
3mL 58/2/40 Water/Acetic Acid/ACN

Elution (E)
2mL Methanol – 2% Acetic Acid

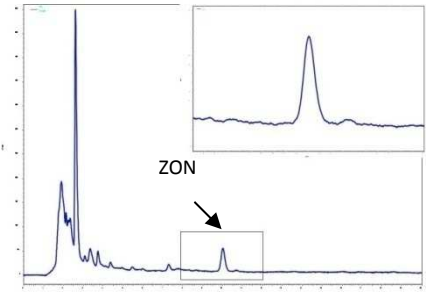
The elution fraction was then evaporated and dissolved in water before HPLC analysis.

HPLC Method with Fluorescence detection
Column: Hypersil Gold C18 - 150mmx 4.6mm
Mobile phase: water/MeOH (40/60, v/v)
Flow rate: 1mL/min
Fluorescence detection: excitation/emission wavelengths: 275 / 450nm
Injection volume: 100µL.

RESULTS



Chromatogram obtained after Cleanup of Maize (contamined at 41 µg / kg) with AFFINIMIP® SPE Zearalenone



Chromatogram obtained after Cleanup of Rice (contamined at 41 µg / kg) with AFFINIMIP® SPE Zearalenone .

Recoveries of Zearalenone at a contamination level of 41µg / kg after AFFINIMIP® SPE Zearalenone . Clean-up in Maize (n=9)

Recoveries %	% RSD
86	8

Catalog number: FS100-02

ZEARALENONE IN CEREAL-BASED BABY FOOD

Regulations for processed cereal based food for baby food:
Europe (EC 1126/2007) : 20µg/Kg

PROTOCOL OF CLEANUP

Sample preparation

Cleanup with a 3mL/100mg AFFINIMIP® SPE Zearalenone cartridge

25g of ground cereal-based samples were extracted with 100 mL of acetonitrile/deionized water (75/25, v/v) for 3 min. The extract was filtered through a folded filter paper and 10 mL of the filtrate were diluted with 10 mL of deionized water. Then, this solution was filtered through a filter paper.
This solution was used as the loading solution.

Equilibration
3mL Acetonitrile
3mL Water

Loading
12mL of loading solution (eq. 1.5g sample)

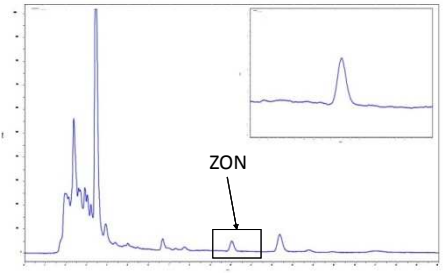
Washing of interferences (W1)
3mL 58/2/40 Water/Acetic Acid/ACN

Elution (E)
2mL Methanol – 2% Acetic Acid

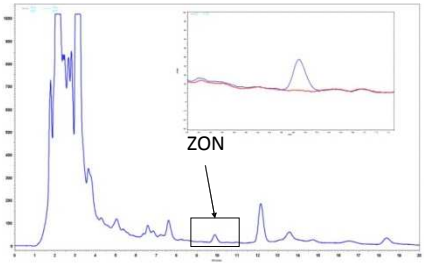
The elution fraction was then evaporated and dissolved in water before HPLC analysis.

HPLC Method with Fluorescence detection
Column: Hypersil Gold C18 -150mm x 4.6mm
Mobile phase: water/MeOH (40/60, v/v)
Flow rate: 1mL/min
Fluorescence detection: excitation/emission wavelengths: 275 / 450nm
Injection volume: 100µL.

RESULTS



Chromatogram obtained after Cleanup of Cereal-based babyfood (contamined at 41µg / kg) AFFINIMIP® SPE Zearalenone (after dilution by 2 of the elution fraction with water).



Chromatograms obtained after Cleanup of Cereal-based babyfood (contamined at 10µg/kg (blue) or 0µg/kg (red)) with AFFINIMIP® SPE Zearalenone (after evaporation of the elution fraction and dissolution in 1mL of the mobile phase).

Recoveries of Zearalenone at a contamination level of 41µg / kg after AFFINIMIP® SPE Zearalenone . Clean-up in Cereal – based baby food (n=5)

Recoveries %	% RSD
80	3

Catalog number: FS100-02

ZEARALENONE IN EDIBLE CORN OIL

Regulations for processed cereal based food for baby food:
Europe (EC 1126/2007) : 20µg/Kg

PROTOCOL OF CLEANUP

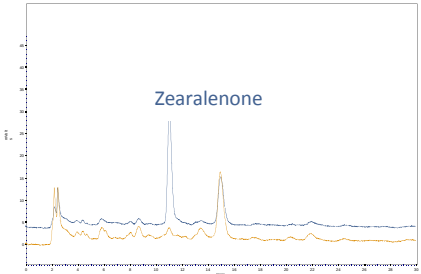
Sample preparation
Corn oil is diluted 1/3 in Diethyl Ether to obtain the loading solution.

Cleanup with a 3mL/100mg AFFINIMIP® SPE Zearalenone cartridge

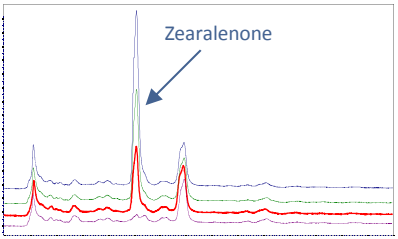
Equilibration
3mL Diethyl Ether
Loading
3mL of loading solution (eq. 1mL of corn oil)
Washing of interferences (W1)
6mL Diethyl ether
Drying 30 seconds
Washing of interferences (W2)
6mL 58/2/40 Water/Acetic Acid/ACN
Elution (E)
4mL Methanol – 2% Acetic Acid
The elution fraction was then evaporated and dissolved in water before HPLC analysis.

HPLC Method with Fluorescence detection same as p 15

RESULTS



Chromatograms of Corn Oil spiked with Zearalenone at 400µg/L (blue) or not spiked (orange) obtained after cleanup by AFFINIMIP®SPE Zearalenone.



Chromatograms obtained after cleanup by AFFINIMIP®SPE Zearalenone of Corn Oil spiked with Zearalenone at 200µg/L (red), 400µg/L (green), 600 µg/L (blue) or not spiked (purple).

Recoveries of Zearalenone in Corn Oil at various contamination levels after AFFINIMIP®SPE Zearalenone cleanup.

C° (µg/L)	Mean C° (µg/L)	Recoveries %
200	230	115
400	440	110
600	678	113

Catalog number: FS100-02

ZEARALENONE IN MEAT

PROTOCOL OF CLEANUP

Sample preparation

25g of meat are mixed during 2 minutes with 100mL of a solution of deionized water/ acetonitrile (50/50). Then the mixture is filtered through filter paper (4-7µm). This solution was then diluted 10 times with deionized water to obtain the loading solution used for the clean-up protocol

Purification with a 3mL/100mg AFFINIMIP® SPE ZEARALENONE cartridge

Equilibration

2mL Acetonitrile

2mL Water

Loading solution

Up to 6mL of loading solution

Washing of interferences

6mL 40/60 Acetonitrile/deionized Water **Drying 3-5min**

Elution (E)

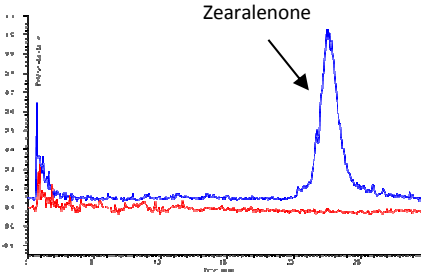
2mL 2/98 Acetic Acid/Methanol

The elution fraction was then concentrated and diluted to mobile phase before HPLC analysis.

RESULTS

Recovery for ZON > 80%*

*** Tested at 960µg/kg**



SIM Chromatograms obtained after clean-up of MEAT with AFFINIMIP® SPE ZEARALENONE.

- Blue trace for spiked with 960µg/kg of ZEARALENONE
- Red trace for the blank sample

HPLC Method with LC-MS

HPLC Column: Hypersil gold column (50mm x 2.1mm)

Mobile phase: 73/27 0,1 Formic acid in water / Acetonitrile

Flow rate: 0.2mL/min

Injection volume: 20µL.

Catalog number: FS100-02

OCHRATOXIN A IN CEREALS

Regulations for unprocessed cereals:
Europe (EC 1881/2006) : 5µg/Kg
Codex Alimentarius Standard: 5µg/Kg for raw wheat

PROTOCOL OF CLEANUP

Sample preparation

50g of finely ground wheat are mixed during 1 minute in a blender with 100mL of extraction solvent (60/40 Acetonitrile/deionized Water). The extract is filtered through a filter paper.

Then, 5mL of the extract is diluted with 5mL of HCl solution pH=1, 0.1M. After a filtration through a filter paper, this solution is used as the loading solution.

Cleanup with a 3mL/100mg AFFINIMIP® SPE Ochratoxin A cartridge

Equilibration
3mL Acetonitrile
3mL Water

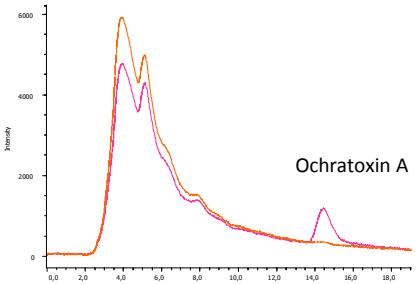
Loading
4mL of loading solution (eq. 1g wheat)

Washing of interferences
6mL 60/40 HCl solution pH 1, 0.1M/ACN

Elution (E)
2mL Methanol – 2% Acetic acid

The elution fraction was then evaporated and dissolved in water before HPLC analysis.

RESULTS



Chromatogram obtained after Cleanup of wheat (spiked at 5µg / kg (pink) or not contaminated (orange)) with AFFINIMIP® SPE Ochratoxin A

Recoveries of Ochratoxin A after AFFINIMIP® SPE Ochratoxin A Clean-up in wheat (n=6)

C° (µg/kg)	Recoveries %	% RSD
5	96.3	7.7

HPLC Method with Fluorescence detection

Column: Hypersil Gold C18 column 150mm x 2.1mm

Mobile phase: water/acetic acid/MeOH (39/1/60, v/v)

Flow rate: 0.2mL/min

Fluorescence detection: excitation/emission wavelengths: 333 / 460nm

Injection volume: 20µL.

Catalog number: FS101-02

OCHRATOXIN A IN PAPRIKA

Regulations for paprika:
Europe (EC 594/2012) : 30µg/Kg until 31.12.14 then 15µg/Kg

PROTOCOL OF CLEANUP

Sample preparation

10g of paprika are shaken during 30 minutes with 100mL of $NaHCO_3$ 1% in water. The extract is centrifuged for 30 minutes at 4000 rpm at room temperature then filtered through a filter paper.

25mL of the extract is diluted with 25mL of HCl solution pH=1, 0.1M. After a filtration through a filter paper, this solution is used as the loading solution.

Cleanup with a 3mL/100mg AFFINIMIP® SPE Ochratoxin A cartridge

Equilibration
3mL Acetonitrile
3mL Water

Loading
4mL of loading solution (eq. 1g sample)

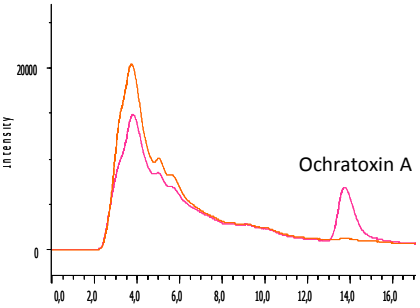
Washing of interferences
6mL 60/40 HCl solution pH 1, 0.1M/ACN

Elution (E)
2mL Methanol – 2% Acetic acid

The elution fraction was then evaporated and dissolved in water before HPLC analysis.

HPLC Method with Fluorescence detection same as p 18

RESULTS



Chromatogram obtained after Cleanup of paprika (spiked at 30µg / kg (pink) or not contaminated (orange)) with AFFINIMIP® SPE Ochratoxin A

Recoveries of Ochratoxin A after AFFINIMIP® SPE Ochratoxin A Clean-up in paprika (n=4).

C° (µg/kg)	Recoveries %	% RSD
30	93.3	3.4

Catalog number: FS101-02

OCHRATOXIN A IN RED AND WHITE WINE

Regulations for wine:
Europe (EC 1881/2006) : 2µg/L

PROTOCOL OF CLEANUP

Sample preparation

10mL of wine is diluted with 10mL of HCl solution pH=1, 0.1M. This solution is used as the loading solution.

Cleanup with a 3mL/100mg AFFINIMIP® SPE Ochratoxin A cartridge

Equilibration
3mL Acetonitrile
3mL Water

Loading
2 to 10mL of loading solution (eq. 1 to 5mL sample)

Washing of interferences
6mL 60/40 HCl solution pH 1, 0.1M/ACN

Elution (E)
2mL Methanol – 2% Acetic acid

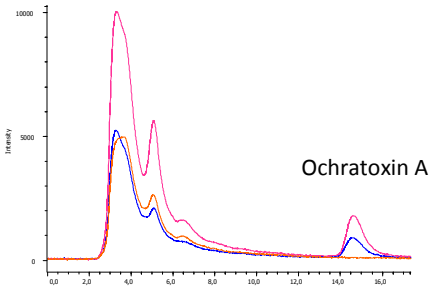
The elution fraction was then evaporated and dissolved in water before HPLC analysis.

Recoveries of Ochratoxin A after AFFINIMIP® SPE Ochratoxin A Clean-up in wine (white and red).

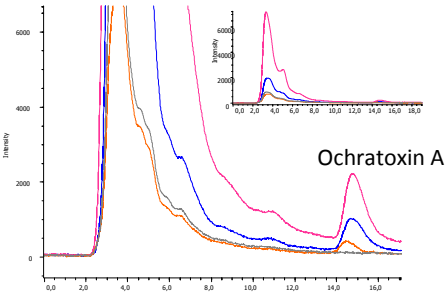
Matrix	C° (µg/kg)	Recovery %	% RSD
White wine (n=10)	2	91.3	6.2
Red wine (n=4)	2	78.8	2.8

HPLC Method with Fluorescence detection same as p 18

RESULTS



Chromatograms obtained after Cleanup of white wine spiked at 2µg/kg (loading with 5mL (blue); loading with 10mL (pink)) and after a loading of 5mL of not contaminated white wine (orange) with AFFINIMIP® SPE Ochratoxin A



Chromatograms obtained after Cleanup of red wine spiked at 2µg / kg (loading with 2mL (orange); loading with 5mL (blue); loading with 10mL (pink)) and after a loading of 2mL of not contaminated red wine (grey) with AFFINIMIP® SPE Ochratoxin A

Catalog number: FS101-02

PATULIN IN BABY FOOD APPLE JUICE

Regulations for apple juice:
Europe (EC 1881/2006) : 50µg/Kg
USA (FDA CPG Sec.510.150) : 50µg/Kg
Regulations for apple juice for infants and young children:
Europe (EC 1881/2006) : 10µg/Kg

PROTOCOL OF CLEANUP

Sample preparation

Loading solution: 2.5mL apple juice and 2.5mL of water-2% acetic acid are mixed.

Cleanup with a 3mL/100mg AFFINIMIP® SPE Patulin cartridge

Equilibration
2mL Acetonitrile
1mL water

Loading
4mL of loading solution

Washing of interferences (W1)
1mL $NaHCO_3$
2mL Water

Drying by applying vacuum 10 seconds

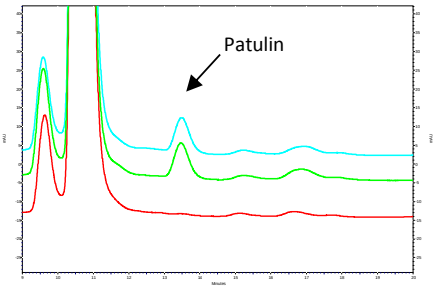
Washing of interferences (W2)
1mL Diethyl Ether

Elution (E)
2mL Ethyl Acetate

The elution fraction was then evaporated and dissolved in water containing 0.1% acetic acid before HPLC analysis.

HPLC Method
Column: Atlantis T3, 150mm x 2.1mm
Mobile phase: Deionized water/ACN (95/5, v/v) Flow rate: 0.2mL/min
Detection: UV - 276nm
Injection volume: 100µL.

RESULTS



Chromatograms obtained after AFFINIMIP® SPE Patulin Clean-up of an apple juice spiked at 10µk/kg with Patulin (Green and blue) or not spiked (Red)

Recovery of Patulin (n=9) at a contamination level of 10µg/kg in apple Juice after AFFINIMIP® SPE Patulin Clean-up.

Recoveries % (n=9)	% RSD_R
97.9	11

Catalog number: FS102-02

PATULIN IN APPLE JUICE

Regulations for apple juice:
Europe (EC 1881/2006) : 50µg/Kg
USA (FDA CPG Sec.510.150) : 50µg/Kg
Regulations for apple juice for infants and young children:
Europe (EC 1881/2006) : 10µg/Kg

PROTOCOL OF CLEANUP

Sample preparation

Loading solution: 2.5mL apple juice and 2.5mL of water-2% acetic acid are mixed.

Clean-up with a 3mL/100mg AFFINIMIP® SPE Patulin cartridge

Equilibration
2mL Acetonitrile
1mL water

Loading
4mL of loading solution

Washing of interferences (W1)
1mL $NaHCO_3$ in Water
2mL Water

Drying by applying vacuum 10 seconds

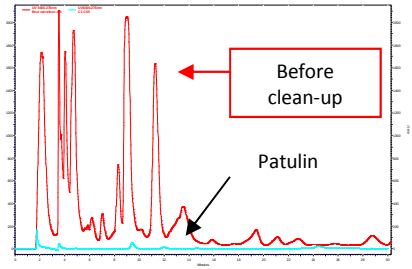
Washing of interferences (W2)
1mL Diethyl Ether

Elution (E)
2mL Ethyl Acetate

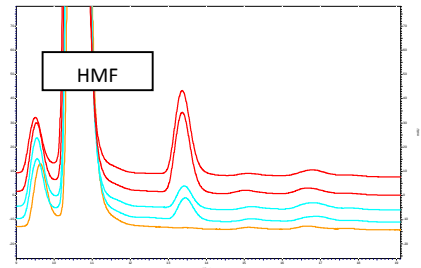
The elution fraction was then evaporated and dissolved in water containing 0.1% acetic acid before HPLC analysis.

HPLC Method same as p 21.

RESULTS



Chromatograms of apple juice containing 25µg/kg of Patulin before (Red) and after (Blue) AFFINIMIP® SPE Patulin Clean-up



Chromatograms obtained after AFFINIMIP® SPE Patulin Clean-up of an apple juice spiked at 40µg/kg (tested twice, red) or at 10µg/kg (tested twice, blue) with Patulin or not spiked (orange)

Recovery of Patulin in apple juice after AFFINIMIP® SPE Patulin Clean-up and relative standard deviation calculated from results generated under reproducibility conditions.

C° of Patulin (ng/mL)	Recovery %	% RSD_R
10	97.9	11 (n=9)
40	90.6	11 (n=41)

Catalog number: FS102-02

PATULIN IN APPLE JUICE USING A SPE AUTOMATE

PROTOCOL OF CLEANUP

Sample preparation

10 mL of turbid or clear apple juice are mixed with 10mL 2% acetic acid and shaked about 30 s with a vortex mixer and shaken about 10 s by hand. The mixture is centrifuged during 10 minutes at 8 000 rpm at RT and filtered . Then it is centrifuged at 10 000 rpm and at RT a second time. A portion of the supernatant (at least 5 mL) is used for the automate ASPEC XLi ™ immediately carefully without disturbing the sediment transferred into a graduated test tube.

Clean-up with a 3mL/100mg AFFINIMIP® SPE Patulin cartridge

Equilibration (1mL/min)

2mL Acetonitrile

1mL water

Loading (0.5mL/min)

4mL of loading solution

Washing of interferences (W1)(2mL/min)

1mL 1% $NaHCO_3$ in Water

2mL Water

Elution (E)

2mL Acetonitrile (0.8mL/min) + 1mL ACN (4mL/min)

The elution fraction was added to 0.5mL 0.1% Acetic acid.

HPLC Method

Column: Gemini C18 column, 150mm x 2mm, 3µm
Mobile phase: gradient

Time (min)	% water	% ACN
0	98	2
11	98	2
11.01	5	95
28	5	95
28.01	98	2
40	98	2

Flow rate: 0.2mL/min
Detection: MS/MS
Injection volume: 25µL.

RESULTS

Validation for apple juice	Average Recoveries %	LoD
5-50µg/Kg	81	8µg/mL

Publications

Data extracted from the article: Maria Barricelli, Deutsche Lebensmittel-Rundschau : DLR ; Analytik, Forschung, Prozesse, Recht Vol. 110, No. 7 (2014), p. 310-315

Catalog number: FS102-02

PATULIN IN BABY FOOD APPLE PUREE

Regulations for apple puree:
Europe (EC 1881/2006) : 25µg/Kg
Regulations for apple puree for infants and young children:
Europe (EC 1881/2006) : 10µg/Kg

PROTOCOL OF CLEANUP

Sample preparation
10g of apple puree, 150µL of a pectinase enzyme solution and 10mL water are mixed. Leave solution at room temperature overnight or for 2h at 40°C. Centrifuge at 4500g for 5min and then filter the solution with a 0.2µm filter. This solution is used as the loading solution.

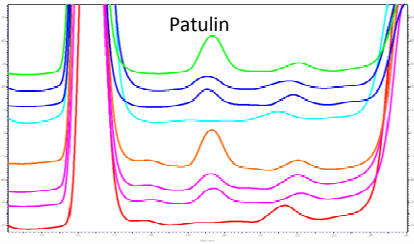
Cleanup with a 3mL/100mg AFFINIMIP® SPE Patulin cartridge

Equilibration
2mL Acetonitrile
1mL Water
Loading
5mL of loading solution
Washing of interferences (W1)
4mL Water -1%Acetic acid
1mL $NaHCO_3$ 1% solution
3mL Water
Drying by applying vacuum 10 seconds
Washing of interferences (W2)
500µL Diethyl Ether
Elution (E)
2mL Ethyl Acetate

The elution fraction was then evaporated and dissolved in water containing 0.1% acetic acid before HPLC analysis.

Catalog number: FS102-02

RESULTS



Chromatograms obtained after AFFINIMIP® SPE Patulin Clean-up of different apple puree.
Clean-up of an apple puree from a well-known brand spiked at 25µg/kg (orange), 10µk/kg with Patulin (pink, tested twice) or not spiked (red).
Clean-up of an apple puree second well known brand spiked at 25µg/kg (green), 10µk/kg with Patulin (dark blue, tested twice) or not spiked (light blue).

Recovery and repeatability of Patulin (n=4) at a contamination level of 10µg/kg in apple puree after AFFINIMIP® SPE Patulin Clean-up.

Recovery % (n=4)	% RSD_R
81.2	2.1

HPLC Method
Column: Atlantis T3 column, 150mm x 2.1mm
Mobile phase: gradient

Time (min)	% water	% ACN
0	98	2
20	98	2
21	50	50
25	50	50
26	98	2

Flow rate: 0.2mL/min
Detection: UV - 276nm
Injection volume: 100µL.

PATULIN IN APPLE PUREE

Regulations for apple puree:
Europe (EC 1881/2006) : 25µg/Kg
Regulations for apple juice for infants and young children:
Europe (EC 1881/2006) : 10µg/Kg

PROTOCOL OF CLEANUP

Sample preparation

10g of apple puree, 150µL of a pectinase enzyme solution and 10mL water are mixed. Leave solution at room temperature overnight or for 2h at 40°C. Centrifuge at 4500g for 5min and then filter the solution with a 0.2µm filter. This solution is used as the loading solution.

Cleanup with a 3mL/100mg AFFINIMIP® SPE Patulin cartridge

Equilibration
2mL Acetonitrile
1mL Water

Loading
5mL of loading solution

Washing of interferences (W1)
4mL Water -1%Acetic acid
1mL $NaHCO_3$ 1% solution
3mL Water

Drying by applying vacuum 10 seconds

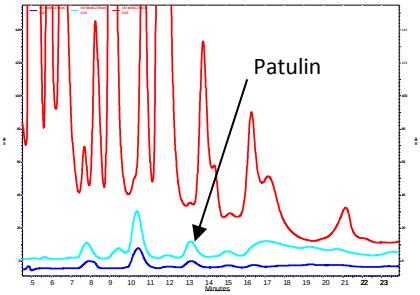
Washing of interferences (W2)
500µL Diethyl Ether

Elution (E)
2mL Ethyl Acetate

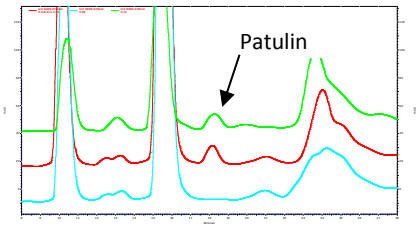
The elution fraction was then evaporated and dissolved in water containing 0.1% acetic acid before HPLC analysis.

HPLC Method same as previous page

RESULTS



Chromatograms of apple puree containing 40µg/kg or 80µg/kg of Patulin before (Red) and after (Blue) AFFINIMIP® SPE Patulin Clean-up



Chromatograms of apple puree containing 0µg/kg (blue) or 20µg/kg (tested twice, green and red)) of Patulin after AFFINIMIP® SPE Patulin Clean-up

Recovery and repeatability of Patulin (n=3) at a contamination level of 20µg/kg in apple puree after AFFINIMIP® SPE Patulin Clean-up.

C° of Patulin (µg/kg)	Recovery % (n=3)	% RSDr
20	84	4.5

Catalog number: FS102-02

PATULIN IN APPLE PUREE – format 6mL

A format tailored for the larger liquid volume required for apple puree protocol

Regulations for apple puree:
Europe (EC 1881/2006) : 25µg/Kg
Regulations for apple juice for infants and young children:
Europe (EC 1881/2006) : 10µg/Kg

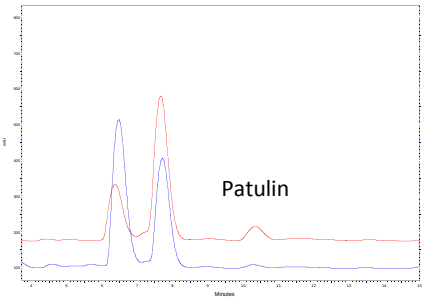
PROTOCOL OF CLEANUP

Sample preparation
10g of apple puree, 150µL of a pectinase enzyme solution and 10mL water are mixed. Leave solution at room temperature overnight or for 2h at 40°C. Centrifuge at 4500g for 5min and then filter the solution with a 0.2µm filter. This solution is used as the loading solution.

Cleanup with a 6mL/200mg AFFINIMIP® SPE Patulin cartridge

Equilibration
2mL Acetonitrile
1mL Water
Loading
5mL of loading solution
Washing of interferences (W1)
4mL Water -1%Acetic acid
1mL $NaHCO_3$ 1% solution
3mL Water
Drying by applying vacuum 10 seconds
Washing of interferences (W2)
500µL Diethyl Ether
Elution (E)
2mL Ethyl Acetate
The elution fraction was then evaporated and dissolved in water containing 0.1% acetic acid before HPLC analysis.

RESULTS



Chromatograms of apple puree spiked with 20µg/kg of Patulin (Red) and not spiked (blue) after AFFINIMIP® SPE Patulin Clean-up

Recovery and repeatability of Patulin (n=6) at a contamination level of 10µg/kg in apple puree after AFFINIMIP® SPE Patulin Clean-up.

C° of Patulin (µg/kg)	Rec. %	% RSDr
10 (n=6)	90	9
20 (n=3)	92	11

HPLC Method same as p 24

Catalog number: FS102-02B-200mg

PATULIN IN APPLE – FRUIT PUREE

Regulations for apple puree:
Europe (EC 1881/2006) : 25µg/Kg
Regulations for apple puree for infants and young children:
Europe (EC 1881/2006) : 10µg/Kg

PROTOCOL OF CLEANUP

Sample preparation
10g of apple puree, 150µL of a pectinase enzyme solution and 10mL water are mixed. Leave solution at room temperature overnight or for 2h at 40°C. Centrifuge at 4500g for 5min and then filter the solution with a 0.2µm filter. This solution is used as the loading solution.

Cleanup with a 3mL/100mg AFFINIMIP® SPE Patulin cartridge

Equilibration
2mL Acetonitrile
1mL Water
Loading
5mL of loading solution
Washing of interferences (W1)
4mL Water -1%Acetic acid
1mL $NaHCO_3$ 1% solution
3mL Water
Drying by applying vacuum 10 seconds
Washing of interferences (W2)
500µL Diethyl Ether
Elution (E)
2mL Ethyl Acetate

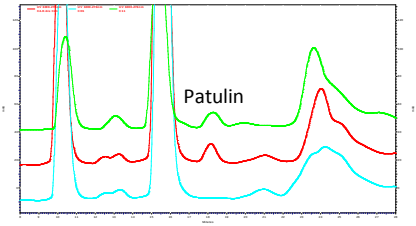
The elution fraction was then evaporated and dissolved in water containing 0.1% acetic acid before HPLC analysis.

HPLC Method
Column: Atlantis T3 column, 150mm x 2.1mm
Mobile phase: gradient

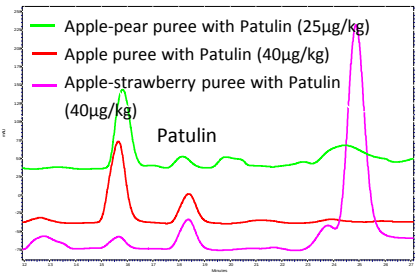
Time (min)	% water	% ACN
0	98	2
20	98	2
21	50	50
25	50	50
26	98	2

Flow rate: 0.2mL/min
Detection: UV - 276nm
Injection volume: 100µL.

RESULTS



Chromatograms of apple puree containing 0µg/kg (blue) or 20µg/kg (tested twice, green and red) of Patulin after AFFINIMIP® SPE Patulin Clean-up.



Chromatograms obtained after AFFINIMIP® SPE Patulin Clean-up of different purees.

Recovery and reproducibility of Patulin with different levels of contamination for all tested apple-fruit puree after AFFINIMIP® SPE Patulin Clean-up.

C° of Patulin (µg/kg)	Recoveries %	% RSD_R
10 (n=9)	77.4	8.1
25 (n=8)	90.9	11.4
40 (n=6)	86.0	11.9

Catalog number: FS102-02

PATULIN IN WHOLE APPLE

Regulations for solid apple products:
Europe (EC 1881/2006) : 25µg/Kg

PROTOCOL OF CLEANUP

Sample preparation

Preparation with microwave

Whole apple is cut into pieces and put in a microwave for 90s before crushing the pieces. 15g sample and 7.5mL water are mixed with 150µL pectinase solution and put overnight at room temperature or for 2h at 40°C before a filtration with filter 4-7µm to obtain the loading solution.

Preparation with a blender

Whole apple is cut into pieces, put in a blender with Water (2:1 Apple: Water) and mix for 1min. 15g sample and 300µL pectinase solution are put overnight at room temperature or for 2h at 40°C before a filtration with filter 4-7µm to obtain the loading solution.

Cleanup with a 3mL/100mg AFFINIMIP® SPE Patulin cartridge

Equilibration
2mL Acetonitrile
1mL Water

Loading
3mL of loading solution

Washing of interferences (W1)
3mL Water-2% Acetic Acid

Drying by applying vacuum 10 seconds

Washing of interferences (W2)
250µL Diethyl Ether

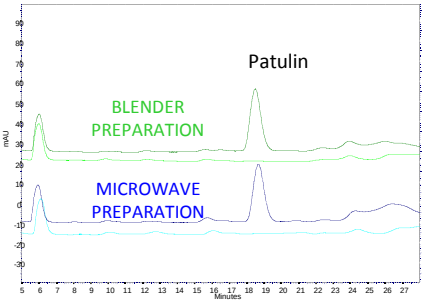
Drying by applying vacuum 10 seconds

Elution (E)
1mL Ethyl Acetate

The elution fraction was then evaporated and dissolved in water containing 0.1% acetic acid before HPLC analysis.

HPLC Method same as p 24

RESULTS



Chromatograms obtained after AFFINIMIP® SPE Patulin Clean-up of whole apple spiked at 40µg/kg with Patulin (dark colors) or not spiked (light colors).

Recovery yields obtained after AFFINIMIP® SPE Patulin Clean-up of spiked whole apple with 40µg/kg of Patulin. Whole apples are prepared according to 2 different methods

Whole apple prepared with blender		Whole apple prepared with microwave	
96	96	95	88

Catalog number: FS102-02

PATULIN IN CIDER

Regulations for cider:
Europe (EC 1881/2006) : 50µg/Kg

PROTOCOL OF CLEANUP

Sample preparation
The cider is degassed by sonicating sample for 1 hour. Then the degas cider is diluted by 2 with water containing 2% of acetic acid. This solution is mixed and used as the loading solution.

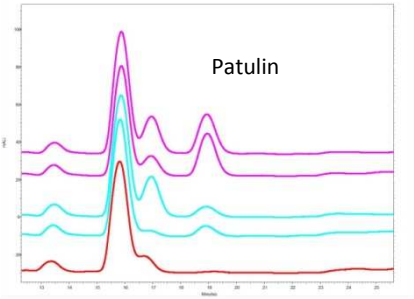
Cleanup with a 3mL/100mg AFFINIMIP® SPE Patulin cartridge

Equilibration
2mL Acetonitrile
1mL Water
Loading
4mL of loading solution
Washing of interferences (W1)
1mL $NaHCO_3$ 1% in Water
2mL Water
Drying by applying vacuum 10 seconds
Washing of interferences (W2)
500µL Diethyl Ether
Elution (E)
2mL Ethyl Acetate

The elution fraction was then evaporated and dissolved in water containing 0.1% acetic acid before HPLC analysis.

HPLC Method
Column: Atlantis T3 column, 150mm x 2.1mm
Mobile phase: Deionized water/ACN (95/5, v/v) Flow rate: 0.2mL/min
Detection: UV - 276nm
Injection volume: 100µL.

RESULTS



Chromatograms obtained after AFFINIMIP® SPE Patulin Clean-up of a cider spiked at 40µg/kg (tested twice, pink) or at 10µg/kg (tested twice, blue) with Patulin or not spiked (red).

Recovery of Patulin at a contamination level of 10µg/kg and 40µg/kg in cider after AFFINIMIP® SPE Patulin Clean-up and relative standard deviation calculated from results generated under reproducibility conditions.

C° of Patulin (ng/mL)	Recoveries %	% RSD_R
10	87.5 (n=2)	-
40	80.5 (n=5)	7.5

Catalog number: FS102-02

PATULIN IN ALCOHOL POMMEAU AND LIQUOR

Regulations for apple based beverage :
Europe (EC 1881/2006) : 50µg/Kg

Manzella liquor contains 20% alcohol and 2.1% of concentrated apple juice.
Alcohol Pommeau is a mixture of Calvados and Apple Juice. It contains 17% Alcohol.

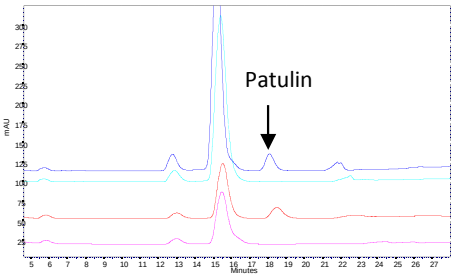
PROTOCOL OF CLEANUP

Sample preparation
To 1mL of Manzella Liquor or Alcohol Pommeau, add 2mL Water to obtain the loading solution.

Cleanup with a 3mL/100mg AFFINIMIP® SPE Patulin cartridge

Equilibration
2mL Acetonitrile
1mL Water
Loading
3mL of loading solution
Washing of interferences (W1)
3mL Water (containing 2% Acetic Acid for AA W1 protocol)
Drying by applying vacuum 10 seconds
Washing of interferences (W2)
250µL Diethyl Ether
Drying by applying vacuum 10 seconds
Elution (E)
2mL Ethyl Acetate
The elution fraction was then evaporated and dissolved in water containing 0.1% acetic acid before HPLC analysis.

RESULTS



Chromatograms obtained after AFFINIMIP® SPE Patulin Clean-up of Manzella liquor spiked at 40µg/L with Patulin (dark blue for Water in W1 and red for Water –AA in W1) or not spiked (light blue and pink). Washing with Acetic acid is more efficient.

Recovery yields obtained for Pommeau and Manzella after AFFINIMIP® SPE Patulin Clean-up. W1 with water or Water - 2%Acetic acid

	Water for W1		Water-AA for W1	
Pommeau	101	101	90	93
Manzella	102	106	87	90

HPLC Method same as p 24

Catalog number: FS102-02

PATULIN IN TOMATO KETCHUP AND TOMATO POWDER

PROTOCOL OF CLEANUP

Sample preparation

Preparation OF TOMATO KETCHUP

10g tomato ketchup and 10mL water are mixed with 150µL pectinase solution and left overnight at RT before a filtration with filter 0.2µm to obtain the loading solution.

Preparation OF TOMATO POWDER

10g tomato ketchup and 20mL water are mixed. 10g of the mixture, 10mL water and 150µL pectinase solution are left overnight at RT before a centrifugation at 4500rpm during 5 min. Then the mixture is filtered with filter 0.2µm to obtain the loading solution.

Cleanup with a 3mL/100mg AFFINIMIP® SPE Patulin cartridge

Equilibration
- 2mL Acetonitrile
- 1mL Water

Loading
- 5mL of loading solution from tomato ketchup or 2mL from tomato powder

Washing of interferences (W1)
- 4mL Water-1% Acetic Acid
- 4mL Water

Drying by applying vacuum 10 seconds

Washing of interferences (W2)
- 500µL Diethyl Ether

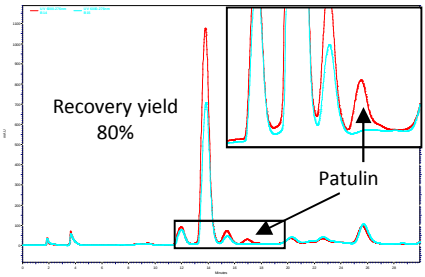
Elution (E)
- 2mL Ethyl Acetate

The elution fraction was then evaporated and dissolved in water containing 0.1% acetic acid before HPLC analysis.

HPLC Method same as p 24

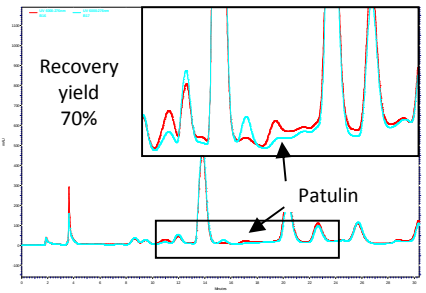
RESULTS

TOMATO KETCHUP



Chromatograms obtained after AFFINIMIP® SPE Patulin Clean-up of TOMATO KETCHUP spiked at 40µg/kg with Patulin (red) or not spiked (light blue).

TOMATO POWDER



Chromatograms obtained after AFFINIMIP® SPE Patulin Clean-up of TOMATO POWDER spiked at 36µg/kg with Patulin (red) or not spiked (light blue).

Catalog number: FS102-02

PATULIN IN BLUEBERRY JUICE

PROTOCOL OF CLEANUP

Sample preparation

5mL Blueberry juice is diluted with 5mL water containing 2% of acetic acid to obtain the loading solution.

Cleanup with a 3mL/100mg AFFINIMIP® SPE Patulin cartridge

Equilibration
- 2mL Acetonitrile
- 1mL Water

Loading
- 4mL of loading solution

Washing of interferences (W1)
- 1mL $NaHCO_3$ 1% in Water
- 2mL Water

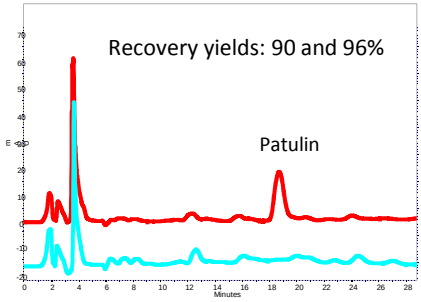
Drying by applying vacuum 10 seconds

Washing of interferences (W2)
- 500µL Diethyl Ether

Elution (E)
- 2mL Ethyl Acetate

The elution fraction was then evaporated and dissolved in water containing 0.1% acetic acid before HPLC analysis.

RESULTS



Chromatograms obtained after AFFINIMIP® SPE Patulin Clean-up of Blueberry juice spiked at 40µg/L with Patulin (red) or not spiked (light blue).

HPLC Method same as p 24

Catalog number: FS102-02

PATULIN IN CONCENTRATE JUICE AND THICK JUICE

PROTOCOL OF CLEANUP

Preparation of fruit juice concentrate samples

2.5g of fruit juice concentrate are mixed with 10mL water and 100µL Pectinase. (REA-001-50mL). Leave the solution at room temperature overnight or for 2h at 40°C. Centrifuge at 4000g for 10min and collect the supernatant. Dilute the supernatant by 2 with Acetic Acid 2% in water. This solution is used as the loading solution.

Preparation of thick fruit juice samples

15mL of thick fruit juice are mixed with 120µL Pectinase (REA-001-50mL). Leave the solution at room temperature overnight or for 2h at 40°C. Centrifuge at 4000g for 10min and collect the supernatant. Dilute the supernatant by 2 with acetic acid 2% in water. This solution is used as the loading solution.

Cleanup with a 6mL/200mg AFFINIMIP® SPE Patulin cartridge

Equilibration
- 4mL Acetonitrile
- 4mL Water

Loading
- 4 to 6mL of loading solution

Washing of interferences (W1)
- 2mL $NaHCO_3$ 1% in Water
- 4mL Water

Drying by applying vacuum 30 seconds

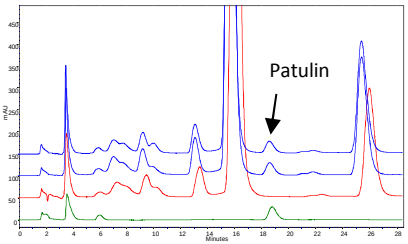
Washing of interferences (W2)
- 1mL Diethyl Ether

Elution (E)
- 2mL Ethyl Acetate

The elution fraction was then evaporated and dissolved in water containing 0.1% acetic acid before HPLC analysis.

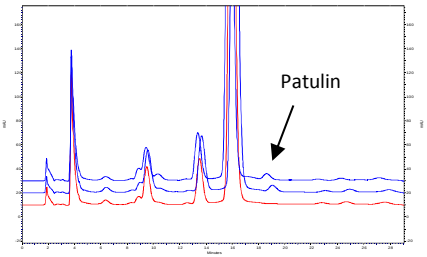
RESULTS

THICK JUICE



Chromatograms obtained after AFFINIMIP® SPE Patulin clean-up of apple mango juice spiked at 20µg/kg (blue) with Patulin or not spiked (red). In green, Patulin solution at 50ng/mL. prepared by dilution of a 100µg/mL Patulin standard solution (REA-PAT-1mL) in mobile phase.

CONCENTRATE JUICE



Chromatograms obtained after AFFINIMIP® SPE Patulin Clean-up of grapefruit juice concentrate spiked at 10µg/kg (blue) with Patulin or not spiked (red).

HPLC Method same as p 24

Catalog number: FS102-02B-200mg

PATULIN IN DRIED APPLE

Regulations for solid apple products:
Europe (EC 1881/2006) : 25µg/Kg

PROTOCOL OF CLEANUP

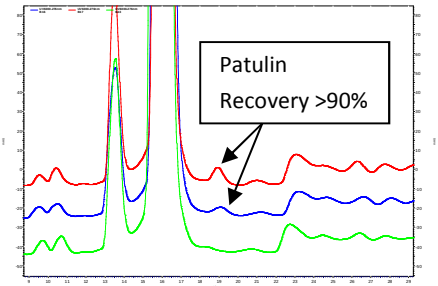
Sample preparation
3g of dried apple dices, 30mL of water and 150µL of pectinase are mixed and left at room temperature overnight. Then, they are centrifuged at 4500rpm during 5min and filtered with 0.2µm filter to obtain the loading solution.

Cleanup with a 6mL/200mg AFFINIMIP® SPE Patulin cartridge

Equilibration
4mL Acetonitrile
2mL Water
Loading
10mL of loading solution
Washing of interferences (W1)
5mL Water-2% Acetic Acid
5mL Water
Drying by applying vacuum 30 seconds
Washing of interferences (W2)
500µL Diethyl Ether
Elution (E)
2mL Ethyl Acetate

The elution fraction was then evaporated and dissolved in water containing 0.1% acetic acid before HPLC analysis.

RESULTS



Chromatograms obtained after AFFINIMIP® SPE Patulin Clean-up of dried apple dices spiked at 20µg/kg (red) or at 10µg/kg (blue) with Patulin or not spiked (green).

HPLC Method same as p 24

Catalog number: FS102-02B-200mg

DEOXYNIVALENOL IN CEREALS FOR FOOD (Water extraction)

Regulations for unprocessed corn or durum wheat for food:
Europe (EC 1126/2007) : 1750µg/Kg

PROTOCOL OF CLEANUP

Sample preparation with **EXTRACTION WITH WATER**

20g of cereals were ground in a blender for 1 minute. Then, 80 ml of deionized water were added. This mixture was then ground for 2 additional minutes. After grinding the mixture was placed in a beaker and left stirred under magnetic agitation for 30 minutes.

Then the whole mixture was transferred in a centrifuge vial and centrifuged at 2500 rpm for 15 minutes. After centrifugation the supernatant was filtered through filter paper. This solution was then diluted 5 times using deionized water.

Cleanup with a 6mL/100mg AFFINIMIP® SPE Deoxynivalenol cartridge

Equilibration
2mL Acetonitrile
2mL Water

Loading
6mL of loading solution

Washing of interferences (W1)
3mL $NaHCO_3$ 1% in water

Drying 30 seconds

Washing of interferences (W2)
1mL Diethylether

Elution (E)
4mL Ethyl Acetate

The elution fraction was then evaporated and dissolved in water -0,1% HCOOH before HPLC analysis.

HPLC Method with MS or UV detection

Column: Hypersil Gold C18 50mm x 2,1mm
Mobile phase: water with 0,1% formic acid/ACN (95/5, v/v)
Flow rate: 0,2mL/min
MS detection: m/z 265 (ESI^-)
UV detection: 220nm
Injection volume: 20µL.

RESULTS

Recovery of Deoxynivalenol after AFFINIMIP®SPE Deoxynivalenol clean-up and relative standard deviation (repeatability conditions).

Matrix	Detection	Mean µg/kg	R%	%RSDr
Corn (800µg/kg)	UV	623.4	78.0	1.4 (n=6)
Corn (800µg/kg)	MS	642.7	80.3	3.4 (n=6)
Wheat (n=3) (600µg/kg)	MS	540.0	90.0	9.8 (n=3)

Catalog number: FS117-02B

DEOXYNIVALENOL IN BABYFOOD CEREALS

Regulations for cereal based food for baby food:
Europe (EC 1126/2007) : 200µg/Kg

PROTOCOL OF CLEANUP

Sample preparation
150 ml of deionized water were added to 20g of cereals - based babyfood. This mixture was then placed in a beaker and left stirring under magnetic agitation for 30 minutes.
Then, the whole mixture was centrifuged at 2500 g for 15 minutes. After centrifugation, the supernatant was filtered through filter paper.

Cleanup with a 6mL/100mg AFFINIMIP® SPE Deoxynivalenol cartridge

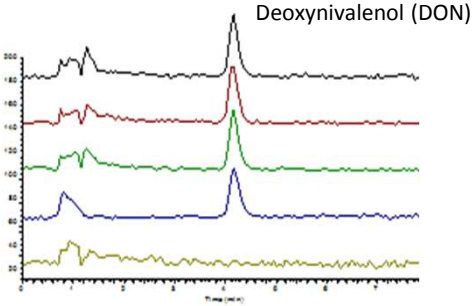
.

Equilibration
2mL Acetonitrile
2mL Water
Loading
6mL of loading solution
Washing of interferences (W1)
3mL $NaHCO_3$ 1% in water
Drying 30 seconds
Washing of interferences (W2)
1mL Diethylether
Elution (E)
4mL Ethyl Acetate

The elution fraction was then evaporated and dissolved in water -0,1% HCOOH before HPLC analysis.

HPLC Method with MS detection same as p35

RESULTS



MS chromatograms obtained after water extraction of Deoxynivalenol from cereals - based babyfoods and clean-up with AFFINIMIP® SPE Deoxynivalenol:

- black, red and green spiked with Deoxynivalenol at 150µg/kg
- dark yellow not spiked
- blue, a standard solution of Deoxynivalenol at 200ng/mL is prepared by dilution of a 100µg/mL Deoxynivalenol standard solution (reference : REA-DON-1mL) in mobile phase

Recovery of Deoxynivalenol after AFFINIMIP®SPE Deoxynivalenol clean-up and relative standard deviation calculated from results generated under reproducibility conditions.

Matrix	C° µg/kg	Mean µg/kg	R%	%RSD R
Babyfood (n=3)	150	136.5	91	0.4

Catalog number: FS117-02B

DEOXYNIVALENOL IN CEREALS FOR ANIMAL FEED

Regulations for DON in animal feed:
Europe (EC 576/2006) :
8mg/Kg for cereals and cereals products
12mg/Kg for maize by-products

PROTOCOL OF CLEANUP

Sample preparation with **EXTRACTION WITH WATER**

20g of animal feed were ground in a blender for 1 minute. Then, 80 ml of deionized water were added. This mixture was then ground for 2 additional minutes. After grinding the mixture was placed in a beaker and left stirred under magnetic agitation for 30 minutes.

Then, the whole mixture was centrifuged at 2500 g for 15 minutes. After centrifugation the supernatant was filtered through filter paper. This solution was then diluted 5 times using deionized water.

Cleanup with a 6mL/200mg AFFINIMIP® SPE Deoxynivalenol cartridge

.

Equilibration
2mL Acetonitrile
2mL Water

Loading
2mL of loading solution

Washing of interferences (W1)
3mL $NaHCO_3$ 1% in water

Drying 30 seconds

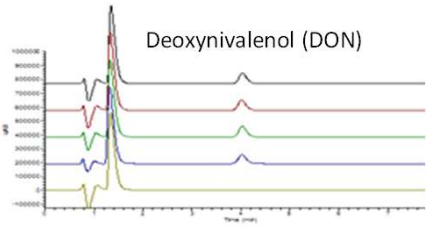
Washing of interferences (W2)
1mL Diethylether

Elution (E)
4mL Ethyl Acetate

The elution fraction was then evaporated and dissolved in water -0,1% HCOOH before HPLC analysis.

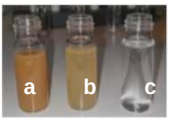
HPLC Method same as p 35

RESULTS



UV chromatograms obtained after WATER extraction of DON from wheat (animal feed) and clean-up with AFFINIMIP®SPE Deoxynivalenol :

- black, red and green spiked with DON at 6mg/kg
- dark yellow not spiked
- blue, a standard solution of DON at 1µg/mL is prepared by dilution of a 100µg/mL Deoxynivalenol standard solution (reference : REA-DON-1mL) in mobile phase



Analysis of Whiskas:
a. Extraction solution with water
b. Loading solution
c. Elution solution

Recovery of Deoxynivalenol after AFFINIMIP®SPE Deoxynivalenol clean-up and relative standard deviation - repeatability conditions (n=3).

Feed Matrices	C° mg/kg	Mean mg/kg	R%	%RSDr
Wheat	6	5.7	94	0.1
Whiskas	0.8	0.73	91	2.4

Catalog number: FS117-02B-200mg

DEOXYNIVALENOL IN MEAT

PROTOCOL OF CLEANUP

Sample preparation

25g of meat are mixed during 2 minutes with 100mL of a solution of deionized water/ acetonitrile (50/50). Then the mixture is filtered through filter paper (4-7µm). This solution was then diluted 10 times with deionized water to obtain the loading solution used for the clean-up protocol

Cleanup with a 6mL/100mg AFFINIMIP® SPE DEOXYNIVALENOL cartridge

Equilibration
2mL Acetonitrile
2mL Water

Loading solution
Up to 6mL of loading solution

Washing of interferences
3mL $NaHCO_3$ 1% solution

Drying 30s

Washing of interferences
1mL Diethyl Ether

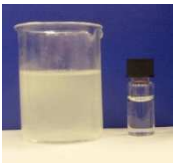
Elution (E)
4mL Ethyl Acetate

The elution fraction was then concentrated and diluted to mobile phase before HPLC analysis.

HPLC Method with MS detection

Column: Hypersil Gold C18 column 50mm x 2,1mm
Mobile phase: water with 0,1% formic acid/ACN (95/5, v/v)
Flow rate: 0,2mL/min
MS detection: m/z 265 (ESI^-)
Injection volume: 20µL.

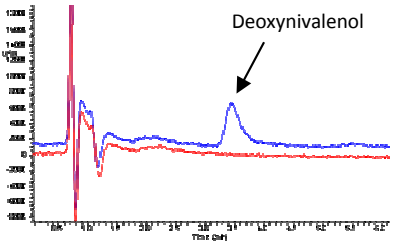
RESULTS



Loading Elution

Recovery for DON > 90%*

*Tested at 8000µg/kg



UV Chromatograms (220nm) obtained after clean-up of MEAT with AFFINIMIP® SPE DEOXYNIVALENOL.

- Blue trace for spiked with 8000µg/kg of DEOXYNIVALENOL
- Red trace for the blank sample

Catalog number: FS117-02B

DEOXYNIVALENOL, 3-AcetylDON AND 15-AcetylDON IN CEREALS (Hydro-organic extraction)

Regulations for unprocessed corn or durum wheat for food:
Europe (EC 1126/2007) : 1750µg/Kg

PROTOCOL OF CLEANUP

Sample preparation **WITH HYDROORGANIC EXTRACTION**

20g of cereals were ground in a blender for 1 minute. Then, a solution of deionized water: acetonitrile (50:50) was added. This mixture was then ground for 2 additional minutes. After grinding, the mixture was placed in a beaker and left stirred under magnetic agitation for 30 minutes.

Then the mixture was centrifuged at 2500 g for 15 minutes. After centrifugation, the supernatant was filtered through filter paper. This solution was then diluted 10 times using deionized water.

Cleanup with a 6mL/100mg AFFINIMIP® SPE Deoxynivalenol cartridge

.

Equilibration
2mL Acetonitrile
2mL Water

Loading
6mL of loading solution

Washing of interferences (W1)
3mL $NaHCO_3$ 1% in water

Drying 30 seconds

Washing of interferences (W2)
1mL Diethylether

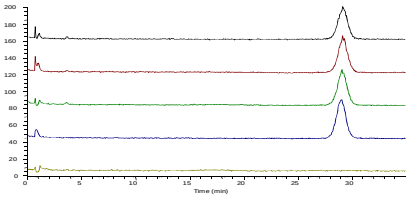
Elution (E)
•4mL Ethyl Acetate

The elution fraction was then evaporated and dissolved in water-0.1% formic acid before HPLC analysis.

HPLC Method with MS detection same as p39 except for the mobile for 3-AcDON and 15-AcDON analyses: water with 0.1% formic acid/ACN (90/10, v/v)

RESULTS

3-AcetylDeoxynivalenol (3-AcDON)



MS chromatograms obtained after hydro-organic extraction of 3-acetylDON from corn and clean-up with AFFINIMIP®SPE Deoxynivalenol :
-black, red and green: spiked with Deoxynivalenol at 800µg/kg
-dark yellow: not spiked
-blue: a standard solution of 3-AcetylDON at 200ng/mL is prepared by dilution of a 100µg/mL 3-AcetylDeoxynivalenol standard solution (reference : REA-3AcDON-1mL) in mobile phase

Recovery obtained for DON, 3-acetylDON and 15-acetylDON after AFFINIMIP®SPE Deoxynivalenol clean-up of Corn and relative standard deviation - repeatability conditions (n=3).

Compound	C° µg/kg	Mean µg/kg	R%	%RSDr
DON	800	653.7	81.7	0.3
3-AcetylDON	800	601.0	75.1	2.3
15-AcetylDON	800	641.8	80.2	3.4

Catalog number: FS117-02B

SIMULTANEOUS DETERMINATION OF MULTIMYCOTOXINS IN WHEAT

WHEAT

Aflatoxin B1, Zearalenone, Ochratoxin A, HT-2, T-2, Fumonisin B1, Deoxynivalenol

PROTOCOL OF CLEANUP

Sample preparation

25g of ground wheat were extracted with 100mL of Acetonitrile/Water (50/50, v/v/v) for 2 min using a blender. The extract was filtered through a folded filter paper and 2mL of the filtrate were diluted with 18mL of water. Then, this solution was filtered through a filter paper.

Cleanup with a 6mL/100mg AFFINIMIP® SPE Multimyco LCMSMS cartridge

Equilibration
3mL Acetonitrile
3mL water

Loading
6mL of loading solution

Washing of interferences (W1)
6mL Water/Acetic acid 1%
3mL Water/Acetonitrile (95/5 v/v)

Drying by applying vacuum 3 minutes

Elution (E)
3mL Methanol/Ethyl Acetate/Formic acid (48.5/48.5/3, v/v/v)

The elution fraction was then evaporated and dissolved in Acetontrile /Water with 0.1% Formic acid (15/85 v/v) before HPLC analysis.

Recovery yield

Recovery of multimycotoxins extracted from wheat and analyzed after AFFINIMIP® SPE Multimyco LCMSMS cleanup

Compound	C° µg/kg	Mean µg/kg	R%
Aflatoxin B1	2	1.6	85
Fumonisin B1	1000	937	94
HT-2	100	119	119
T-2	50	56.5	113
Zearalenone	50	54	108
Deoxynivalenol	1250	1025	82
Ochratoxin A	3	2.6	88

UFLC Method
Column: Phenomenex Kinetix XB-C18
Detection: LC-MS/MS
Injection volume: 20µL.

Catalog number: FS118-02

FUMONISINS B1 / B2 AND ZEARALENONE IN MAIZE-BASED BABY FOOD

Regulations for maize-based baby food:
Zearalenone
Europe (EC 1126/2007) : 20µg/Kg
Fumonisins
Europe (EC 1126/2007) : 200µg/Kg

PROTOCOL OF CLEANUP

Sample preparation

Cleanup with a 3mL/100mg AFFINIMIP® SPE FumoZON cartridge

25g of ground samples were extracted with 100 mL of Acetonitrile/Methanol/deionized Water (25/25/50, v/v/v) for 3 min using a blender. The extract was filtered through a folded filter paper and 10 mL of the filtrate were diluted with 10 mL of deionized water. Then, this solution was filtered through a filter paper.
This solution was used as the loading solution.

Equilibration
2mL Acetonitrile
2mL Water

Loading
6mL of loading solution

Washing of interferences
6mL 60/40 Water/ACN

Elution (E)
2mL Methanol – 2% Acetic Acid

The elution fraction was then evaporated and dissolved in water before HPLC analysis.

HPLC Method with MS detection

Column: Hypersil Gold C18 column 50mm x 2.1mm
Mobile phase ZON AND FB1: Water-Formic Acid 0.1%/ACN (73/27)
Mobile phase FB2: Water-Formic Acid 0.1%/ACN (65/35)
Flow rate: 0.2mL/min
MS detection: m/z 722 for Fumonisin B1 (ESI^+)
m/z 706 for Fumonisin B2 (ESI^+)
m/z 317 for Zearalenone (ESI^-)
Injection volume: 20µL.

RESULTS

Recovery of Zearalenone, Fumonisins B1 and B2 in maize-based baby food after AFFINIMIP® SPE FumoZON clean-up and relative standard deviation calculated from results generated under reproducibility conditions.

Sample	C° µg/kg	Mean µg/kg	Recoveries %	% RSD_R
Zearalenone	20	16.9	84.4	1.6 (n=4)
Fumonisin B1	200	168.6	84.3	1.4 (n=3)
Fumonisin B2	200	185.6	92.8	1.9 (n=3)

ION SUPPRESSION EVALUATION

Ion suppression phenomenon can induce an erroneous quantification. To evaluate the ion-suppression, blank maize-based baby food samples were cleaned up with AFFINIMIP® SPE FumoZON . The SPE extracts were spiked with a mixture of Fumonisin B1 and Zearalenone at 2 different concentrations. The standard calibration curves were compared to the matrix SPE extracts. The use of AFFINIMIP® SPE FumoZON strongly reduces ion-suppression phenomena with a maximum of 15% observed for Fumonisins.

Ion suppression percentage obtained in Maize-based baby food (tested twice).

Analyte	C° µg/kg	Ion suppression %
Zearalenone	10	1% and 5%
Zearalenone	50	0% and 5%
Fumonisin B1	100	8% and 11%
Fumonisin B1	500	12% and 14%

Catalog number: FS109-02

FUMONISINS B1 / B2 AND ZEARALENONE IN MAIZE FLOUR

Regulations for cereal flour:
Zearalenone
Europe (EC 1126/2007) : 75µg/Kg
Fumonisins
Europe (EC 1126/2007) : 1000µg/Kg for maize flour
USA: FDA advisory 2000µg/Kg

PROTOCOL OF CLEANUP

Sample preparation

Cleanup with a 3mL/100mg AFFINIMIP® SPE FumoZON cartridge

25g of ground samples were extracted with 100 mL of Acetonitrile/Methanol/deionized Water (25/25/50, v/v/v) for 3 min using a blender. The extract was filtered through a folded filter paper and 10 mL of the filtrate were diluted with 10 mL of deionized water. Then, this solution was filtered through a filter paper.

This solution was used as the loading solution.

Equilibration
2mL Acetonitrile
2mL Water

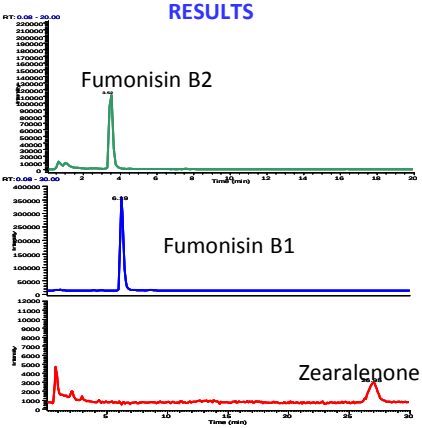
Loading
6mL of loading solution

Washing of interferences
6mL 60/40 Water/ACN

Elution (E)
2mL Methanol – 2% Acetic Acid

The elution fraction was then evaporated and dissolved in water before HPLC analysis.

RESULTS



Chromatograms obtained after AFFINIMIP® SPE FumoZON Clean-up of a maize flour spiked at 38µg/kg with Zearalenone, 2408µk/kg with Fumonisin B1 and 630µg/kg with Fumonisin B2.

Recovery of Zearalenone, Fumonisins B1 and B2 in maize flour after AFFINIMIP® SPE FumoZON clean-up and relative standard deviation calculated from results generated under reproducibility conditions

Sample	C° µg/kg	Mean µg/kg	Yield %	% RSD_R
ZON	38	39.2	103.2	8.5 (n=8)
Fumonisin B1	2408	2002.2	83.1	10.3 (n=8)
Fumonisin B1	400	401.0	100.2	- (n=2)
Fumonisin B2	630	684.6	108.7	11.5 (n=3)

HPLC Method with MS detection same as p 41

Catalog number: FS109-02

Affinity-based SPE sorbents have been developed to be selective in extracting the target analytes like molecularly imprinted polymer (MIP) and immunoaffinity sorbent.
Immunoaffinity columns (IAC) are biological sorbents based on the use of antibodies that are specific to the target analytes.
Molecularly imprinted polymer is a synthetic material with artificially generated three-dimensional network able to specifically rebind a target molecule.
Based on molecularly imprinted polymers, AFFINISEP's AFFINIMIP® SPE cartridges have the advantages to be highly selective and specific. Contrary to IAC, AFFINIMIP® SPE cartridges are chemically and thermally stable, compatible with all solvents as well as cost effective.

PROPERTIES OF MIP AND IAC

Feature	IAC	AFFINIMIP® SPE
Selectivity	High	High
Capacity	6µmol/g	10-100µmol/g
Analyte recognition in water	Good	Variable
Analyte recognition in Organics	Poor	Good
Stability	Poor	Very High
Reproducibility	Variable	Good
Cost	Expensive	Inexpensive

Compared to IAC, AFFINIMIP® SPE provides:
Easier and faster protocol
Lower dilution
Easier automatisation

(Cf. Automated method for the selective SPE of Ochratoxin A from wheat Using Molecularly Imprinted Polymer; Gilson Application Notes Handbook 2011; volume 1 Issue 4)

PROTOCOL: Zearalenone (ZON) from maize flour

Step	Vicam IAC	AFFINIMIP®SPE ZON
Extraction of target analyte	25g sample in 100mL 90/10 Methanol/water Blender 3 minutes + filtration	25g sample in 100mL 75/25 ACN/water Blender 3 minutes + filtration
Preparation loading solution	4mL extract + 96mL water	10mL extract + 10mL Water
Loading	100mL Loading solution	8mL Loading solution
Washing	20mL Water	4mL 2/58/40 Acetic acid / water / ACN
Elution	1.5mL Methanol	2mL 98/2 Methanol/Acetic acid
Protocol time	55min	30min

PROTOCOL: Ochratoxin A (OTA) from wheat flour

Step	Vicam IAC	AFFINIMIP®SPE OTA
Extraction of target analyte	50g sample in 100mL 60/40 ACN/water Blender 1 minute + filtration	
Preparation loading solution	10mL extract + 40mL PBS	10mL extract + 10mL HCl 0.1M pH=1
Loading	10mL Loading solution	4mL Loading solution
Washing	10mL PBS 10mL Water	7mL 60/40 HCl 0.1M pH=1/ACN
Elution	1.5mL Methanol	2mL 98/2 Methanol/Acetic acid
Protocol time	30min	20min

CHROMATOGRAM ASPECT → Equivalent chromatograms

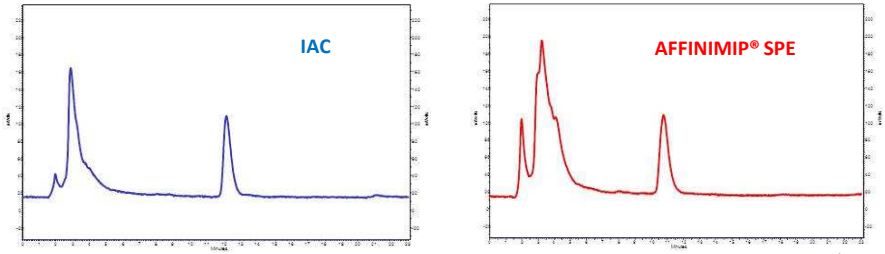


Figure 1. Chromatogram of Maize sample spiked with Zearalenone at 85 µg/kg obtained after cleanup by AFFINIMIP®SPE Zearalenone (red) or Vicam IAC (blue).

RECOVERIES → Higher Recoveries obtained with AFFINIMIP® SPE

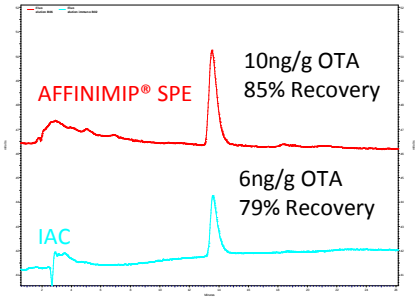


Figure 2. Chromatogram of wheat sample spiked with Ochratoxin A obtained after cleanup by AFFINIMIP®SPE Zearalenone (red, spiked at 10ng/g) or Vicam IAC (blue, spiked at 6ng/g).

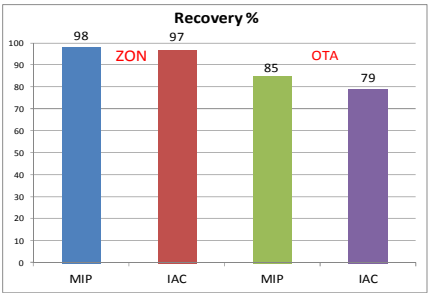


Figure 3. Recovery of Ochratoxin A or Zearalenone obtained after cleanup by AFFINIMIP®SPE or Vicam IAC.

CAPACITY → Capacity MIP > Capacity IAC

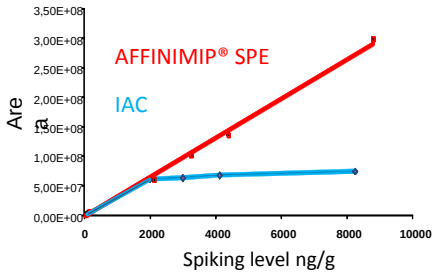


Figure 4. Comparison of capacity between AFFINIMIP®SPE Zearalenone (red) and Vicam IAC (blue).

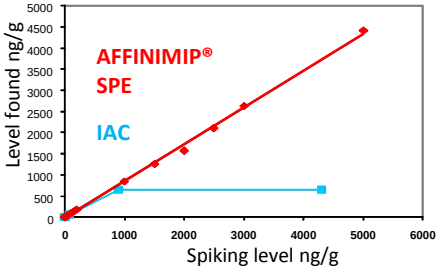


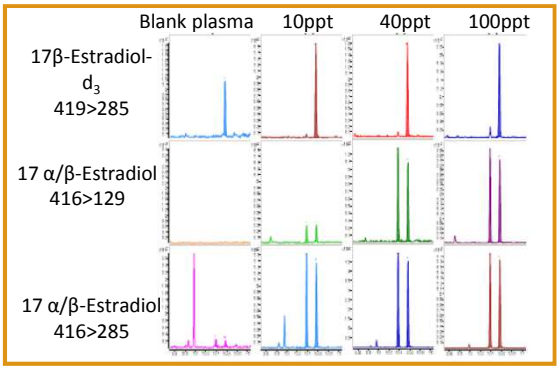
Figure 5. Comparison of capacity between AFFINIMIP®SPE OTA (red) and Vicam IAC (blue).

Analysis of ENDOCRINE DISRUPTING COMPOUNDS

ESTROGENS IN PLASMA

Regulations for Estrogens:
Europe (EC directive) : 40pg/mL of plasma or serum of bovine animals

RESULTS



MRM chromatograms from GC-MS/MS analysis of fortified calves' plasma samples at 0, 10, 40 and 100 pg.mL^{-1} with 17α-estradiol, 17β-estradiol and estrone. Chromatograms obtained after a clean-up with AFFINIMIP® SPE Estrogens *(Courtesy of Emmanuelle Bichon - LABERCA)*

PROTOCOL OF CLEANUP

Sample preparation

2mL serum samples spiked with 40pg 17β-Estradiol-d3. Then 2mL of Acetate buffer (0.8M, pH 6.8) and 100µL β-glucuronidase were added. Hydrolysis performed overnight at 37°C and samples centrifuged at 4000 rpm for 10min. Upper layer was used as loading solution.

Cleanup with a 3mL/100mg AFFINIMIP® SPE Estrogens cartridge

Equilibration
- 3mL Methanol
- 3mL Acetonitrile
- 3mL Water

Loading solution from sample preparation

Washing of interferences
- 3mL Water
- 3mL Water/Acetonitrile (60/40)

Elution (E)
- 3mL Methanol

The elution fraction was then evaporated and estrogens were derivatised 40min at 60°C with BSTFA before GC-MS/MS analysis.

GC-MS/MS Analysis

Column: RTX-1614 Resteck 15m x 0.25mm x 0.10µm

Gradient temperature: 80 to 320°C (15°C/min)

Data extracted from 'Quantification of estrogens at ppt levels in bovine plasma by Molecularly Imprinted Solid Phase Extraction and GC-MS/MS analysis', Emmanuelle Bichon *et al.* (LABERCA) Poster session, HTSP-2 and HTC 2012

Catalog number: FS104-02

SYNTHETIC AND NATURAL ESTROGENS IN RIVER WATER

PROTOCOL OF PURIFICATION

Sample preparation

100mL of river water were filtered through 0.45µm cellulose filter to obtain the loading solution.

Purification with a 3mL/100mg AFFINIMIP® SPE Estrogens cartridge

Equilibration
5mL Acetonitrile
5mL Water

Loading solution from sample preparation

Washing of interferents
4mL Water/Acetonitrile (80/20)
2mL Water

Drying under vacuum during 5min

Washing of interferents
2mL Acetonitrile
2mL Methanol/Acetonitrile (5/95)

Elution (E)
3mL Methanol

The elution fraction was then evaporated and reconstituted in 500µL of UHPLC.

LC-MS Analysis

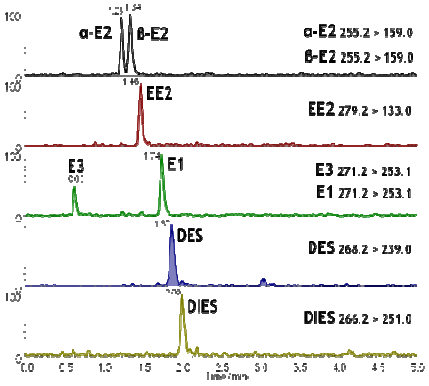
Column: Ascentis Express Phenyl-Hexyl 150mmx2.1mm, 2.7µm

Column Temperature: 35°C

Mobile phase: Water/Acetonitrile/Methanol (51/44/5) at 450µL/min

Catalog number: FS104-02

RESULTS



SRM Chromatograms of Estrogens extracted from 100 mL river water spiked at 100 ng L^{-1} (Courtesy of P. Lucci, University of Barcelona, SPAIN)

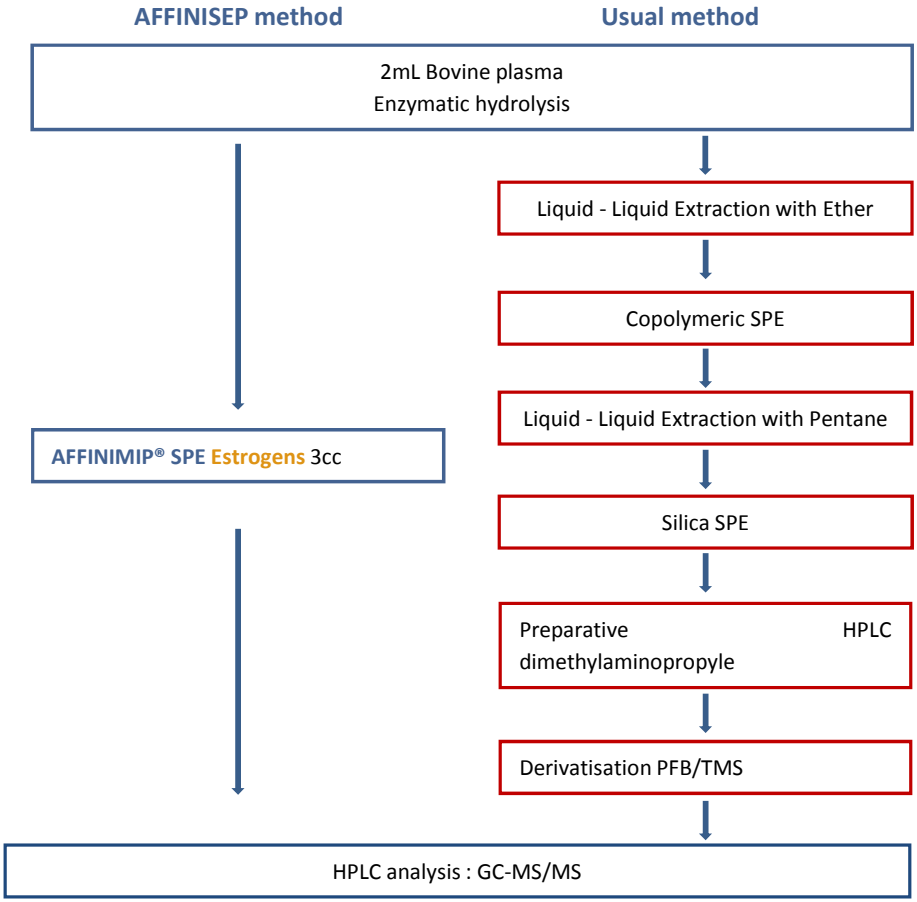
Recovery yield in river water

Matrix	Recovery %
Estrone (E1)	89
17α-Estradiol (α-E2)	101
17β-Estradiol (β-E2)	93
Estriol (E3)	82
17α- Ethynilestradiol (EE2)	100

Publications

Data extracted from **Solid-phase extraction using molecularly imprinted polymer for selective extraction of natural and synthetic estrogens from aqueous samples,** Paolo Lucci, Oscar Núñez, M.T. Galceran, *Journal of Chromatography A, 1218(30), 4828-4833, 2011*

PROTOCOL COMPARISON – AFFINIMIP® SPE ESTROGENS vs usual protocol



Performance. Save your time.

•Data extracted from 'Quantification of estrogens at ppt levels in bovine plasma by Molecularly Imprinted Solid Phase Extraction and GC-MS/MS analysis', Emmanuelle Bichon *et al.* (LABERCA) Poster session, HTSP-2 and HTC 2012

Catalog number: FS104-02

BISPHENOL A IN LIQUID INFANT FORMULA

Regulations for Bisphenol A:
Europe (directive 2011/8/EU) : forbiden in infant feeding bottles

PROTOCOL OF CLEANUP

Sample preparation

Cleanup with a 3mLor 6mL/100mg AFFINIMIP® SPE Bisphenols cartridge

Equilibration
- 3mL Methanol -2% Acetic Acid
- 3mL Acetonitrile
- 3mL Water

Loading
- Up to 15mL of infant formula (pH adjusted to 5-6)

Washing of interferences
- 9mL Water
- 6mL Water/Acetonitrile (60/40)

Drying 30 seconds

Elution (E)
- 3mL Methanol

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

HPLC Method with Fluorescence detection

Column: Hypersil Gold C18 column 150mm x 4.6mm

Mobile phase: gradient profile

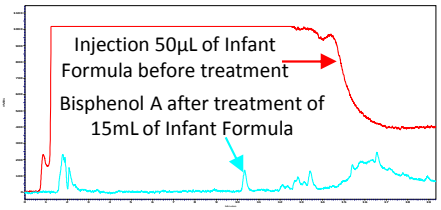
Time (min)	% water	% ACN
0	65	35
2	65	35
12	50	50
20	50	50
20.5	65	35
35	65	35

Flow rate: 1mL/min

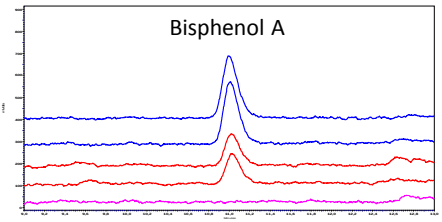
Fluorescence detection: excitation/emission wavelengths: 230 / 315nm

Injection volume: 50µL.

RESULTS



Chromatograms of Infant Formula containing 1µg/L of Bisphenol A before clean-up (Red) and after clean-up (Blue) with AFFINIMIP® SPE Bisphenols.



Chromatograms obtained after clean-up with AFFINIMIP® SPE Bisphenols of 15mL of Infant Formula spiked with Bisphenol A at 2µg/L (tested twice, blue) or at 1µg/L (tested twice, red) or not spiked (pink).

Recovery of Bisphenol A in 15mL of infant formula after AFFINIMIP® SPE Bisphenols clean-up and relative standard deviation calculated from results generated - under **reproducibility** conditions **% RSDR**

[illegible] (µg/L)	Mean (µg/L)	Recoveries %	% RSD_R
1.0	0.8	84.4	7.4
2.0	1.7	85.8	5.3

Catalog number: FS106-02

BISPHENOL A IN POWDERED INFANT FORMULA

Regulations for Bisphenol A:
Europe (directive 2011/8/EU) : forbiden in infant feeding bottles

PROTOCOL OF CLEANUP

Sample preparation

4.4g powdered infant milk was reconstituted in 30 mL of water and warmed up at ~ 50°C during 20 seconds using microwaves. Then 20 mL of acetonitrile were added to 20 mL of warm milk and centrifuged at 4000 rpm during 10 minutes. The supernatant was collected and filtered on filter paper (4-7µm). This extract was diluted 1:1 with water to form the loading solution.

Cleanup with a 3mL or 6mL/100mg AFFINIMIP® SPE Bisphenols cartridge

Equilibration
- 3mL Methanol -2% Acetic Acid
- 3mL Acetonitrile
- 3mL Water

Loading
- Up to 40mL of infant formula (pH adjusted to 5-6)

Washing of interferences
- 9mL Water
- 6mL Water/Acetonitrile (60/40)

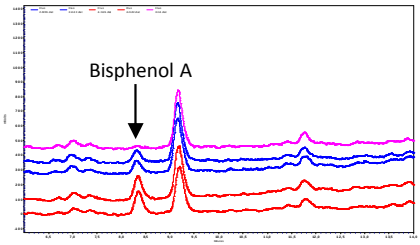
Drying 30 seconds

Elution (E)
- 3mL Methanol

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

HPLC Method with Fluorescence detection same as p 49

RESULTS



Chromatograms obtained after clean-up with AFFINIMIP® SPE Bisphenols of equivalent at 10mL of Infant Formula spiked with Bisphenol A at 4.3µg/L (tested twice, red) or at 2.1µg/L (tested twice, blue) or not spiked (pink).

Recovery of Bisphenol A spiked at different concentrations after 3mL/100mg AFFINIMIP® SPE Bisphenols clean-up of 40mL of loading solution (equivalent to 10mL of reconstituted Infant milk) and relative standard deviation calculated from results generated under repeatability conditions

C° of BPA in reconstituted milk (µg/L)	Mean concentration (µg/L)	Recovery %	RSD_r %
2.1	2.3 (n=5)	108	8.7
4.3	4.0 (n=4)	95	3.7

Catalog number: FS106-02

PROTOCOL COMPARISON – AFFINIMIP® SPE Bisphenols vs competitor

POWDERED INFANT FORMULA ANALYSIS

AFFINISEP method

Precipitation (15g sample + 15mL ACN)
Centrifuge and collect supernatant

↓

Dilute 20mL supernatant with 20mL H_20
Load on **AFFINIMIP® SPE Bisphenols** 3cc (40mL)
Wash 10mL H_20 and 6mL 40% ACN ;
Elute 3mL 100% MeOH

↓

HPLC analysis :
LC Fluorescence detector
or GC-MS/MS or LC-MS/MS

WATERS method*

Precipitation (10g sample + 10mL ACN)
Centrifuge and collect supernatant

↓

Quecher 1
Add contents from DisQue tube 1.
Shake. Centrifuge and collect 10mL

↓

Quecher 2
Add contents from DisQue tube 2.
Shake. Centrifuge and collect supernatant

↓

Dilute supernatant with 70mL H20
Load on **SPE cartridge OASIS HLB** 3cc (70mL)
Wash 2mL 40% Methanol ;
Elute 1mL 100% MeOH

↓

HPLC analysis : LC-MS/MS

•Extract from Waters application note, published 2012 : Rapid analysis of Bisphenol A

Performance. Save your time.

Catalog number: FS106-02

BISPHENOL A IN CANNED FOOD (Liquid form)

Regulations for Bisphenol A:
Europe (directive 2011/8/EU) : Specific migration limit in food from packaging of 0.6mg/kg

PROTOCOL OF CLEANUP

Cleanup with a 3mL or 6mL/100mg AFFINIMIP® SPE Bisphenols cartridge

Equilibration
3mL Methanol -2% Acetic Acid
3mL Acetonitrile
3mL Water

Loading
10mL liquid from canned food after filter paper filtration (pH adjusted to 5-6)

Washing of interferences
9mL Water
6mL Water/Acetonitrile (60/40)

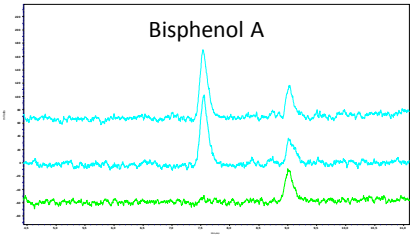
Drying 30 seconds

Elution (E)
3mL Methanol

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

HPLC Method with Fluorescence detection same as p 49

RESULTS



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

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 repetability conditions (n=4).

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

- under reproducibility conditions (n=4).

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

More information in the application note on our website

Catalog number: FS106-02

BISPHENOL A IN CANNED FOOD (Vegetable)

Regulations for Bisphenol A:
Europe (directive 2011/8/EU) : Specific migration limit in food from packaging of 0.6mg/kg

PROTOCOL OF CLEANUP

Sample preparation

150g of drained canned peas - carrots and 200mL of Water /ACN (50/50) are blended during 2 min and centrifuged during 10min at 4000rpm. The supernatant solution is collected , filtered (4-7µm) and diluted ½ with water to give the loading solution. (pH adjusted to 5-6)

Cleanup with a 3mL or 6mL/100mg AFFINIMIP® SPE Bisphenols cartridge

Equilibration
- 3mL Methanol -2% Acetic Acid
- 3mL Acetonitrile
- 3mL Water

Loading
- 20mL loading solution

Washing of interferences
- 9mL Water
- 6mL Water/Acetonitrile (60/40)

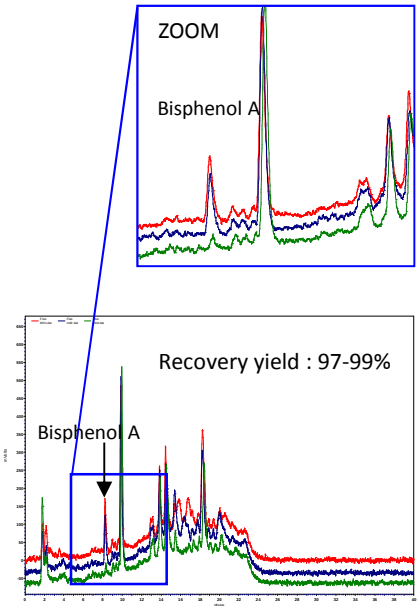
Drying 30 seconds

Elution (E)
- 3mL Methanol

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

HPLC Method with Fluorescence detection same as p 49

RESULTS



Chromatograms after clean-up with AFFINIMIP® SPE Bisphenols of 20mL loading solution of extract of canned Peas- carrots spiked with Bisphenol A at 2µg/L (tested twice, blue and red) or not spiked (green).

Catalog number: FS106-02

BISPHENOL A IN BEER

Regulations for Bisphenol A:
Europe (directive 2011/8/EU) : Specific migration limit in food from packaging of 0.6mg/kg

PROTOCOL OF CLEANUP

Sample preparation
The beer is degassed by sonication for 1 hour. (pH adjusted to 5-6)

Cleanup with a 3mL or 6mL/100mg AFFINIMIP® SPE Bisphenols cartridge

Equilibration
3mL Methanol -2% Acetic Acid
3mL Acetonitrile
3mL Water

Loading
10mL of degassed beer

Washing of interferences
9mL Water
6mL Water/Acetonitrile (60/40)

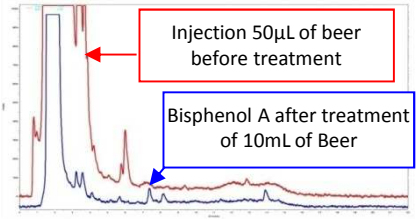
Drying 30 seconds

Elution (E)
3mL Methanol

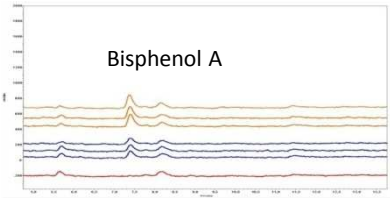
The elution fraction was then evaporated and dissolved in the mobile phase before HPLC analysis.

HPLC Method with Fluorescence detection **same as p 49**

RESULTS



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



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)

Recovery of Bisphenol A in spiked beer after AFFINIMIP® SPE Bisphenols clean-up and relative standard deviation calculated from results generated under reproducibility conditions (**% RSDR**).

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

Catalog number: FS106-02

BISPHENOL A IN RED/WHITE WINES

Regulations for Bisphenol A:
Europe (directive 2011/8/EU) : Specific migration limit in food from packaging of 0.6mg/kg

PROTOCOL OF CLEANUP

Cleanup with a 3mL or 6mL/100mg AFFINIMIP® SPE Bisphenols cartridge

Equilibration
3mL Methanol -2% Acetic Acid
3mL Acetonitrile
3mL Water

Loading
Up to 10mL of wine (pH adjusted to 5-6)

Washing of interferences
9mL Water
6mL Water/Acetonitrile (60/40)

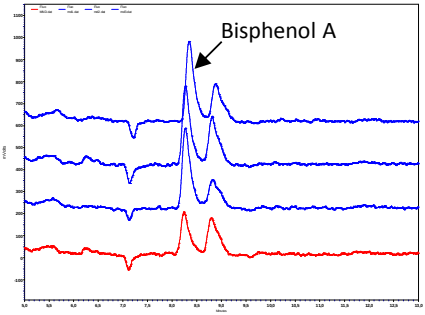
Drying 1 minute

Elution (E)
3mL Methanol

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

HPLC Method with Fluorescence detection same as p 49

RESULTS



Chromatograms obtained after clean-up with AFFINIMIP® SPE Bisphenols of 10mL of white wine spiked with Bisphenol A at 2µg/kg (tested three times, blue) or not spiked (red). The white wine naturally contained 2µg/kg of BPA

Recovery of Bisphenol A spiked at 2µg/kg after AFFINIMIP® SPE Bisphenols clean-up of 6mL of red wine or 10mL of white wine.

Matrice Spiked at 2µg/kg	Mean C° (µg/kg)	Recoveries %
Red wine 1	1.93 (n=2)	96.6
Red wine 2	2.13 (n=2)	106.5
Red wine 3	1.66 (n=2)	83.0
White wine	1.60 (n=3)	80.0

Catalog number: FS106-02

BISPHENOL A IN COLA DRINKS

PROTOCOL OF CLEAUNP

Cleanup with a 3mL or 6mL/100mg AFFINIMIP® SPE Bisphenols cartridge

Equilibration
3mL Methanol -2% Acetic Acid
3mL Acetonitrile
3mL Water

Loading
6mL of Cola drinks after 30min degassing with ultrasounds (pH adjusted to 5-6)

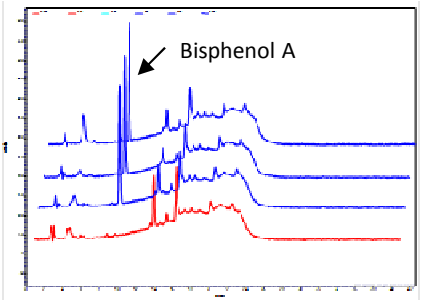
Washing of interferences
9mL Water
6mL Water/Acetonitrile (60/40)

Drying 3 minute

Elution (E)
3mL Methanol

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

RESULTS



Chromatograms obtained after clean-up with AFFINIMIP® SPE Bisphenols of 10mL of white wine spiked with Bisphenol A at 2µg/kg (tested three times, blue) or not spiked (red). The white wine naturally contained 2µg/kg of BPA

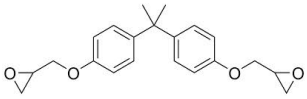
Recovery of Bisphenol A spiked at 5µg/kg after AFFINIMIP® SPE Bisphenols clean-up of 6mL of Cola drinks

Mean concentration (µg/kg)	Recoveries %	RSDr %
1.93 (n=2)	96.6	1.0

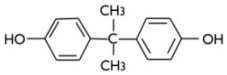
HPLC Method with Fluorescence detection same as p 49

Catalog number: FS106-02

BISPHENOL A AND BADGE IN MILK



Bisphenol A Diglycidyl Ether (BADGE)



Bisphenol A

PROTOCOL OF CLEANUP

Cleanup with a 3mL or 6mL/100mg AFFINIMIP® SPE Bisphenols cartridge

Equilibration
- 3mL Methanol -2% Formic Acid
- 3mL Acetonitrile
- 3mL Water

Loading
- 9mL of Milk (pH adjusted to 5-6)

Washing of interferences
- 9mL Water
- 6mL Water/Acetonitrile (60/40)

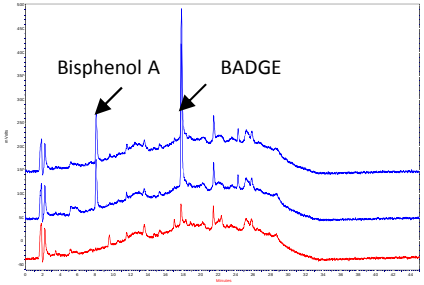
Drying 3 minute

Elution (E)
- 3mL Methanol (E1)
- 3mL Acetonitrile (E2)

The elution fractions E1 and E2 were gathered, evaporated and dissolved in the mobile phase before HPLC analysis.

HPLC Method with Fluorescence detection **same as p 49**

RESULTS



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).

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

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

Catalog number: FS106-02

TOTAL BISPHENOL A IN HUMAN URINE

PROTOCOL OF CLEANUP

Sample preparation

3mL urine sample, 1mL of sodium acetate buffer 0.1M at pH 5.0 and 20µL of β-glucuronidase/sulfatase *Helix pomatia* enzyme solution at 1.0mg/mL in the same buffer were mixed thoroughly by vortex. The enzymatic reaction was carried out for 2h at 37°C to obtain the loading solution.

Cleanup with a 6mL/100mg AFFINIMIP® SPE Bisphenols glass cartridge

Equilibration

3mL Methanol -2% Acetic Acid
3mL Acetonitrile
3mL Water

Loading solution

Up to 12mL of loading solution (Equivalent to around 9mL of urine)

Washing of interferences

4mL Water
4mL Water/Acetonitrile (60/40)

Elution (E)

3mL Methanol

The elution fraction was then concentrated and diluted to 1mL before HPLC analysis.

HPLC Method with LC-MS/MS

HPLC Column: Kinetex 2.6µm PFP 100mm x 4.6mm

Mobile phase: gradient profile

Time (min)	% water	% Methanol
0	70	30
1	70	30
2	5	95
5	5	95
6	70	30
9	70	30

Flow rate: 0.5mL/min

Injection volume: 20µL.

Detector: ESI-MS/MS

RESULTS

Mean percentage recoveries of Bisphenol A spiked at different concentrations in 3mL of urine after AFFINIMIP® SPE Bisphenols clean-up:

C° (ng/mL)	1	10	100
Recoveries %	102.6	94.7	97.6

By courtesy of Nadia Diano, Dept. of Experimental Medicine, Second University of Naples (Italy)

More details in the following article

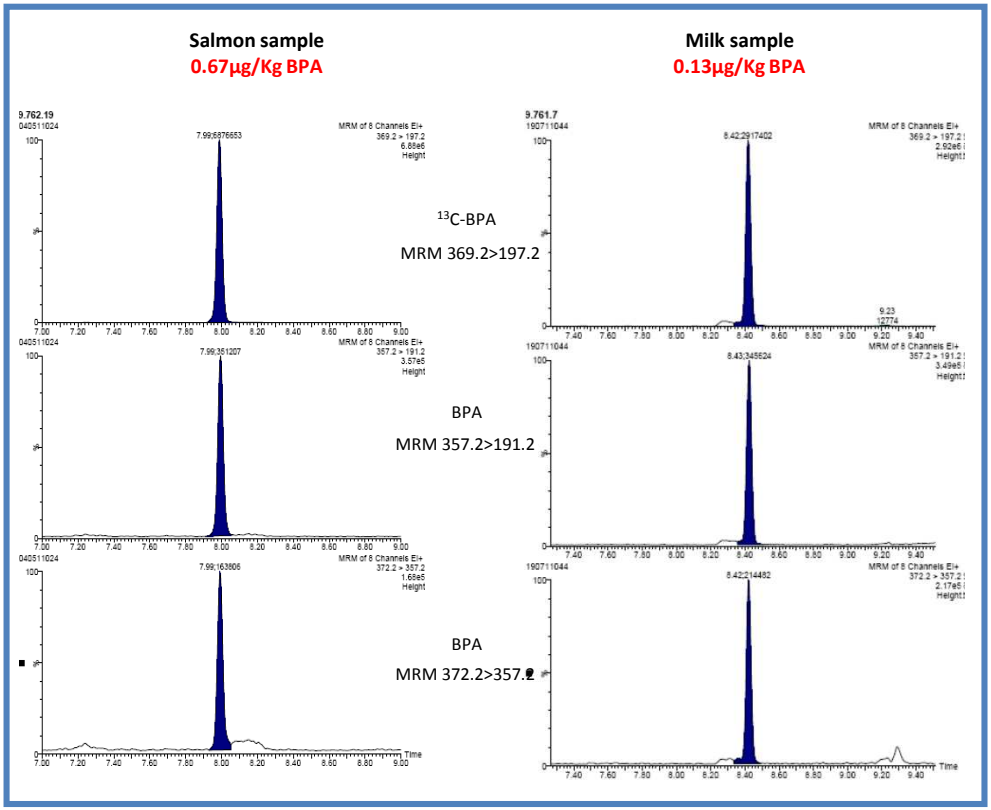
C. Nicolucci, S. Rossi, C. Menale, E. Giudice, P. Miraglia del Giudice, L. Perrone, P. Gallo, D. Mita, N. Diano, *Analytical and Bioanalytical Chemistry*, 1618-2642, 2013.

Catalog number: FS106-02G

BISPHENOL A BY GC-MS/MS

The analysis of BPA (derivatized with TMS) was performed by **GC-MS/MS**, SRM mode after a clean-up protocol using **AFFINIMIP® SPE Bisphenols** of various solid and liquid complex food matrices (illustration here for salmon and milk).

RESULTS



Publications

Data extracted from the poster **Utilisation de la spectrométrie de masse pour le dosage du Bisphénol A dans les matrices alimentaires, Emmanuelle Bichon et al. (LABERCA), Poster for SMAP 2011, Avignon (France)**

Catalog number: FS106-02

FRENCH HEALTH AGENCY REPORT ON BISPHENOL A IN ALL LIQUID AND SOLID FOOD

A report of the French Health Agency (ANSES) on **assessment of the health risks associated with bisphenol A** (BPA) was published on 9 April 2013. Quantitative analysis of Bisphenol A in all liquid or solid food matrices were carried out by using AFFINIMIP® SPE Bisphenols (Analyses carried out by LABERCA and described in Annex 12 of Annexes of the report p132 (in french)).

The analytical method has been described by ONIRIS - LABERCA in the article:
Development and validation of a specific and sensitive gas chromatography tandem mass spectrometry method for the determination of bisphenol A residues in a large set of food items, Y. Deceuninck, E. Bichon, S. Durand, N. Bemrah, Z. Zendong, M.L. Morvan, P. Marchand, G. Dervilly-Pinel J.P., Journal of Chromatography A, 1362, 241-249 (2014)

Example of tested food:
Cereals for breakfast, muesli, cornflakes
Bread, toast, brioche, pastries, sweet and salted biscuits, cookies, pasta…
Cereals: rice, wheat…
Cheese: camenbert, cantal…
Milk (skimmed, concentrated …), Yoghurt, cream, butter
Oils, eggs
Fish: cooked fish, fried breaded fish, canned atun, steamed and smoked salmon, hake…
Seafood: crustacean, oysters, mussel, shrimp…
Vegetable: salad, tomatoes, radish, onion, soja, carrots, cauliflower, zucchini, peas, spinash….
Cooked food such as paella, couscous
Meat: roasted meat, lamb, pork, duck, beef, sheep, turkey, poultry
Delicatessen: Raw and cooked ham, foie gras, paté, sausage, bacon, chipolatas, merguez…
Fruits and dried fruits: almonds, peach, orange, compote….
Drink water, apple juice, soda…
Coffee, chocolate, cacao…

7 BISPHENOL ANALOGS BY LC-MS/MS

The analysis of seven bisphenol analogues in beverage and canned food samples was performed by using **AFFINIMIP® SPE Bisphenols** prior LC–MS analysis.

Bisphenol analogs tested: BPS, BPF, BPA, BPB, BPAF, tetrachlorobisphenol A (TCBPA), TBBPA,.

Matrices : beverage and canned food (soda, tea drink, juice, red wine, vegetable, fish and meat)

PROTOCOL OF PURIFICATION

Sample preparation for beverage

10mL beverage is degassed or centrifuged 9000g during 5min.

Sample preparation for canned food

1g of canned food is extracted with 5mL acetonitrile with sonication during 20min and centrifugation 9000g for 5min. Fat is removed with 5mL Hexane by LLE. The acetonitrile layer is concentrated to 1mL and diluted with water to 10mL

Purification with a 6mL/100mg AFFINIMIP® SPE Bisphenols cartridge

Equilibration
- 5mL Methanol -2% Acetic Acid
- 5mL Acetonitrile
- 5mL Water

Loading
- Loading solution

Washing of interferents
- 6mL Water
- 3mL Water/Acetonitrile (60/40)

Drying 30 min

Washing of interferents
- 2mL Acetonitrile
- 2mL Methanol/Acetonitrile (10/90)

Elution (E)
- 4mL Methanol containing 2% Formic Acid

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

RESULTS FOR CANNED FISH

Analyte	Conc (ng/mL)	Recovery (%)	LOQ (ng/g)
BPS	0.1	73	0.07
	0.5	82	
BPF	1	78	0.5
	5	73	
BPA	0.5	81	0.12
	2.5	89	
BPB	1	79	1.5
	5	82	
BPAF	0.1	81	0.03
	0.5	79	
TCBPA	0.5	72	0.28
	2.5	78	
TBBPA	1	57	0.6
	5	61	

Publications

Data extracted from the article Molecularly imprinted solid phase extraction for the selective extraction of bisphenol analogues in beverages and canned food, Y. Yang et al., *J. Agric. Food Chem.*, 2014, 62 (46), pp 11130–11137

Catalog number: FS106-02B

18 BISPHENOL ANALOGS IN HUMAN BREAST MILK BY GC-MS/MS

ONIRIS – LABERCA describes an accurate and sensitive method of determination of 18 Bisphenol analogues in human breast milk by GC-MS/MS. By using **AFFINIMIP® SPE Bisphenols** in the sample preparation protocol, LABERCA analyzes FREE and TOTAL bisphenol analogues with recovery yields higher than 90% for all analogues.

Analyte	Recovery (%) Spiked at 0.1ng	Recovery (%) Spiked at 1ng	Recovery (%) Spiked at 10ng
Bisphenol A	97	94	105
Bisphenol B	96	99	102
Bisphenol AP	100	90	92
Bisphenol AF	100	96	90
Bisphenol BP	108	109	99
Bisphenol C	92	94	97
Bisohenol CI2	102	101	93
Bisphenol E	96	94	102
Bisphenol PH	94	93	102
Bisphenol S	100	99	93
Bisphenol F	103	109	104
DHDPE	104	92	100
Bisphenol FL	103	100	96
Bisohenol Z	100	97	103
Biphenyl-4,4'-diol	109	103	104
Bisphenol M	96	96	94
Bisphenol P	97	92	99
Bis-2(hydroxyphenyl)methane	108	103	109

Publications

Data extracted from the article Determination of bisphenol A and related substitutes/analogues in human breast milk using gas chromatography-tandem mass spectrometry, Y. Deceuninck, E. Bichon, P. Marchand, C.-Y. Boquien, A. Legrand, C. Boscher, J. P. Antignac, B. Le Bizec, *Anal. and Bioanal. Chem.*, 407 (9), 2485-2497 (2015)

Catalog number: FS106-02

BPA, NONYLPHENOL AND 4-t-OCTYLPHENOL IN FISH FEED

The metabolic effects induced by feed contaminated with a lower or a higher Concentration of **nonylpnenol (NP), 4-tert-octylphenol (t-OP)** or **bisphenol A (BPA)**, three environmental endocrine disruptors, were assessed in juvenile sea bream liver.

The extraction of NP, t-OP and BPA in water and feed was performed by using **AFFINIMIP® SPE Bisphenols** prior LC/ESI-QTRAP-MS/MS analysis.

PROTOCOL OF PURIFICATION

Sample preparation for feed
1g of homogenized feed and 5mL water/Acetonitrile 50/50 were shaken for 10min then centrigated at 1267g for 10min . The supernatant was collected and the extraction on feed was repeated. Then 2mL supernatant and 50µL solution NaCL 20% were mixed with 4mL ethyl acetate, vortexed and centrifuged at 1267g for 5 min. The upper layer was evaporated under nitrogen and diluted xith 2mL Water/Acetonitrile (50/50) and émL water to form the loading solution.

Purification with a 6mL/100mg AFFINIMIP® SPE Bisphenols cartridge

Equilibration
5mL Methanol -2% Acetic Acid
5mL Acetonitrile
5mL Water

Loading
Loading solution

Washing of interferents
10mL Water
6mL Water/Acetonitrile (60/40)

Elution (E)
3mL Methanol

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

Publications

Data extracted from the article Xenobiotic-contaminated diets affect hepatic lipid metabolism: implications for liver steatosis in Sparus aurata juveniles, F. Maradonna, V. Nozzi, S. Santangeli , I. Traversi, P. Gallo, E. Fattore, D.G. Mita, A. Mandich, O. Carnevali, *Aquatic Toxicology,* 257–264 (167), 2015

Catalog number: FS106-02B

PARABENS IN COSMETIC PRODUCTS

PROTOCOL OF CLEANUP

Sample preparation

1g of lotion was mixed 1minute with 1mL of H2SO4 2M and 50mL of 90/10 Ethanol/Water. The mixture was heated during 5min at 60°C. Then the solution is filtered on filter paper (4-7µm). This extract was diluted by 3 with water. The solution was spiked with methylparaben to simulate a concentration of paraben in the lotion at 0.2%, 0.4% and 0.8%.

Cleanup with a 3mL/100mg AFFINIMIP® SPE Phenolics cartridge

Equilibration
3mL Acetonitrile
3mL Water

Loading
Up to 5mL of loading solution

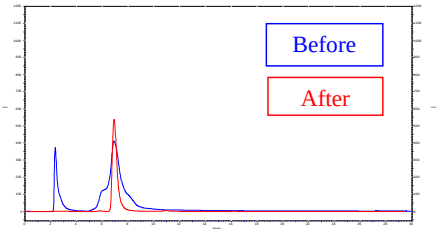
Washing of interferences
3mL Water / Acetonitrile (75/25 v/v)

Elution (E)
3mL Methanol
The elution fraction was diluted by 2 with water prior to analysis.

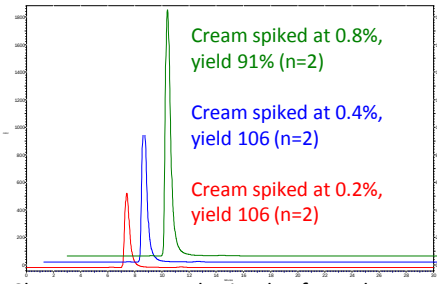
HPLC-UV Method

Column: Thermo Hypersil gold, 150mm x 2.1mm
Mobile phase: 60/40 (v/v) Water/Methanol
Flow rate: 0.2mL/min
Detection: UV - 254nm
Injection volume: 20µL.

RESULTS



Chromatograms of a cream containing 0.2% of methylparaben before clean-up (blue) and after clean-up (Red) with AFFINIMIP® SPE Phenolics.



Chromatograms obtained after clean-up with AFFINIMIP® SPE Phenolics of a cream (without parabens) spiked with different concentrations of methylparaben

Recovery yields and reproducibility after AFFINIMIP® SPE Phenolics Clean-up.

Recoveries % (n=6)	RSD_R%
101.1	8

Catalog number: FS103-02

Analysis of ANTIBIOTICS AND DRUG RESIDUES

CHLORAMPHENICOL IN HONEY

Regulations for Chloramphenicol in residues in food of animal origin:
Europe 2003/181/EC prohibited with a minimum required performance limits of 0.3µg/Kg

PROTOCOL OF CLEANUP

Sample preparation

10g of honey and 10mL Water were mixed under magnetic stirring during 10 minutes and used as the loading solution.

Cleanup with a 1mL/50mg AFFINIMIP® SPE Chloramphenicol cartridge

Equilibration
- 2mL Acetonitrile
- 2mL Water

Loading
- 1mL of loading solution for 15µg/kg (or 10mL for 0.3µg/Kg)

Washing of interferences (W1)
- 1mL Water
- 1mL (Water - 0.5% AA)/ACN (95/5)
- 2mL of Ammonia (1%) in Water
- 2mL (Water-1% Ammonia)/ACN (80/20)

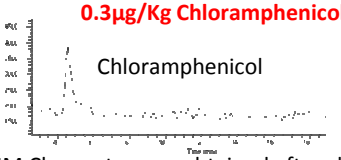
Drying 1 min

Washing of interferences (W2)
- 0.25mL Diethyl ether

Elution (E)
- 2mL Methanol

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

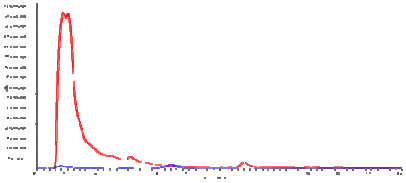
0.3µg/Kg Chloramphenicol



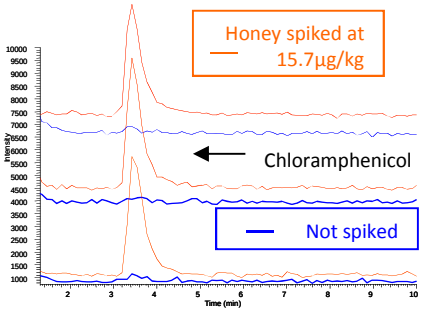
SIM Chromatogram obtained after clean-up with AFFINIMIP® SPE Chloramphenicol of 10g of Honey spiked with Chloramphenicol at 0.3µg/kg.

HPLC Method with MS detection same as p 67

RESULTS



UV Chromatograms obtained after clean-up with AFFINIMIP® SPE Chloramphenicol of 1g of Honey spiked with Chloramphenicol at 15.7µg/kg (red) or not spiked (blue).



SIM Chromatograms obtained after clean-up with AFFINIMIP® SPE Chloramphenicol of 1g of Honey spiked with Chloramphenicol at 15.7µg/kg (red) or not spiked (blue).

Recovery of Chloramphenicol spiked at 16µg/kg after AFFINIMIP® SPE Chloramphenicol clean-up of 1g of Honey and relative standard deviation calculated from results generated under **reproducibility** conditions (**% RSD_R**).

C° (µg/kg)	Mean (µg/kg)	Recoveries %	% RSD_R
15.7	16.9	108.1	6.5 (n=6)
18.2	16.6	91.4	11.4 (n=12)

Catalog number: FS110-02A

CHLORAMPHENICOL IN BOVINE URINE

Regulations for Chloramphenicol in residues in food of animal origin:
Europe (2003/181/EC) : prohibited with a Minimum Required Performance Limits of 0.3µg/Kg
USA FDA: prohibited

PROTOCOL OF CLEANUP

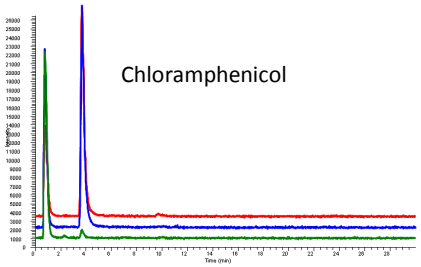
Sample preparation
10 mL of urine were adjusted at pH 7 with Ammonia 1%. This solution was mixed and used as the loading solution.

Cleanup with a 1mL/50mg AFFINIMIP® SPE Chloramphenicol cartridge

Equilibration
2mL Acetonitrile
2mL Water
Loading
1mL of loading solution
Washing of interferences (W1)
1mL (Water - 0.5% Acetic Acid)/Acetonitrile (95/5)
2mL of Ammonia (1%) in Water
2mL (Water-1% Ammonia)/Acetonitrile (80/20))
Drying 1 min
Washing of interferences (W2)
0.25mL Diethyl ether
Elution (E)
2mL Methanol
The elution fraction was then evaporated and dissolved in the mobile phase before HPLC analysis.

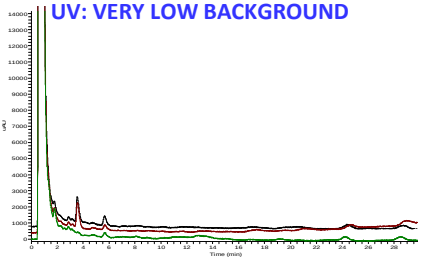
HPLC Method with MS detection
Column: Thermo Accucore C18 column 50mm x 2.1mm
Mobile phase: Ammonium acetate (10mM) in water /Methanol (75/25)
flow rate: 0.2mL/min
MS detection: m/z 321 (ESI^-)
Injection volume: 20µL.

RESULTS



SIM Chromatograms obtained after clean-up with AFFINIMIP® SPE Chloramphenicol of 1 mL of Urine spiked with Chloramphenicol at 17.6µg/kg (red and blue) or not spiked (green).

UV: VERY LOW BACKGROUND



UV Chromatograms of Urine spiked with Chloramphenicol at 17.6 µg/kg (red and black) or not spiked (green) after clean-up with AFFINIMIP® SPE Chloramphenicol

Recovery of Chloramphenicol spiked at 17.6µg/kg after AFFINIMIP® SPE Chloramphenicol clean-up of 1 mL of Urine.

C° (µg/kg)	Mean (µg/kg)	Recovery %
17.6	16.7	90

Catalog number: FS110-02A

CHLORAMPHENICOL IN SHRIMP

Regulations for Chloramphenicol in residues in food of animal origin:
Europe (2003/181/EC) : prohibited with a Minimum Required Performance Limits of 0.3µg/Kg
USA FDA: prohibited

PROTOCOL OF CLEANUP

Sample preparation

5g peeled shrimp were homogenized 2min with a vortex in 20mL of ethyl acetate. Then the solution was filtered on filter paper (25µm). The supernatant was evaporated to dryness and reconstituted in 10mL of Water to obtain the loading solution.

Cleanup with a 1mL/50mg AFFINIMIP® SPE Chloramphenicol cartridge

Equilibration
2mL Acetonitrile
2mL Water

Loading
1 or 2mL of loading solution

Washing of interferences (W1)
1mL Water
1mL (Water - 0.5% Acetic Acid)/Acetonitrile (95/5)
2mL of Ammonia (1%) in Water
2mL (Water-1% Ammonia)/Acetonitrile (80/20))

Drying 1 min

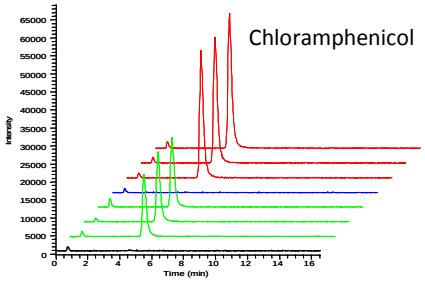
Washing of interferences (W2)
0.25mL Diethyl ether

Elution (E)
2mL Methanol

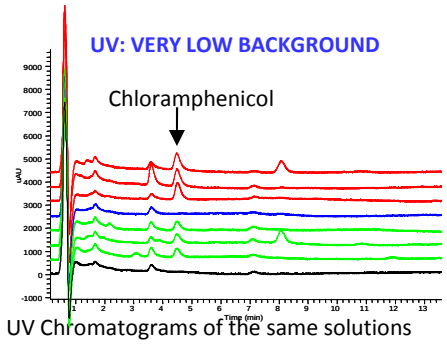
The elution fraction was then evaporated and dissolved in the mobile phase before HPLC analysis.

HPLC Method with MS detection same as p 67

RESULTS



SIM Chromatograms obtained after clean-up with AFFINIMIP® SPE Chloramphenicol of Shrimp spiked with Chloramphenicol at 38µg/kg. Loading of 1mL (spiked in green and not spiked in black) and of 2mL (spiked in red and not spiked in blue)



UV Chromatograms of the same solutions

Recovery of Chloramphenicol spiked at 38µg/kg after AFFINIMIP® SPE Chloramphenicol clean-up of Shrimp.

C° (µg/kg)	Loading volume	Mean (µg/kg)	Recovery %
38	1mL	38.7	101.7
38	2mL	36.4	95.8

Catalog number: FS110-02A

AMPHETAMINES IN HUMAN URINE

Example of Regulations:
France : prohibited cut-off limit of 1µg/mL in urine and 50ng/mL of blood
Virginia (USA): 100ng/mL of blood

PROTOCOL OF CLEANUP

Sample preparation

Human urine is diluted by 2 with an ammonium acetate buffer (13mM, pH 8.5). The pH of the diluted urine is adjusted with NH_3 or CH_3COOH at pH 8.5.

Cleanup with a 3mL AFFINIMIP® SPE Amphetamines cartridge

Equilibration
1mL Acetonitrile
2mL Water

Loading
5mL of diluted urine

Washing of interferences (W1)
3mL Water
3mL Water/Acetonitrile (60/40)

Drying 30 seconds

Elution (E)
1.5mL Methanol – 2% Formic acid

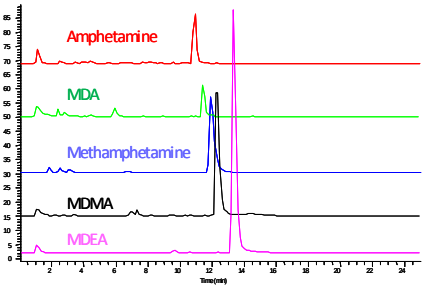
The elution fraction was then evaporated and dissolved in the mobile phase before HPLC analysis.

Capacity: different concentrations of Amphetamine in urine were applied on AFFINIMIP® SPE Amphetamines cartridge (25mg) to measure the capacity of the product.

Quantity loaded µg	Quantity obtained in the elution fraction µg
1.0	0.90
2.5	2.41
5.0	3.51

HPLC Method with MS detection same as p 69

RESULTS



Mass Chromatogram (SIM) obtained after AFFINIMIP® SPE Amphetamines clean-up of a human urine sample spiked at 20ng/mL with Amphetamine and its derivatives.

Recovery of Amphetamines in human urine spiked at 20ng/mL after AFFINIMIP® SPE Amphetamines clean-up and relative standard deviation calculated from results generated under reproducibility conditions.

Sample	Mean ng/mL	Recoveries %	% RSD_R
Amphetamine	17.5	87.5	8.9 (n=8)
MDA	18.6	93.1	9.6 (n=8)
Methamphetamine	18.6	93.2	9.2 (n=8)
MDMA	21.1	105.4	1.5 (n=4)
MDEA	20.3	101.7	12.4 (n=8)

Catalog number: DG102-02

AMPHETAMINES IN HUMAN SERUM

Example of Regulations:
France : prohibited cut-off limit of 1µg/mL in urine and 50ng/mL of blood
Virginia (USA): 100ng/mL of blood

PROTOCOL OF CLEANUP

Sample preparation

Human serum is diluted by 5 with an ammonium acetate buffer (13mM, pH 8.5). The pH of the diluted urine is adjusted with NH_3 or CH_3COOH at pH 8.5.

Cleanup with a 3mL AFFINIMIP® SPE Amphetamines cartridge

Equilibration
1mL Acetonitrile
2mL Water

Loading
2.5mL of diluted serum

Washing of interferences (W1)
3mL Water
3mL Water/Acetonitrile (60/40)

Drying 30 seconds

Elution (E)
1.5mL Methanol – 2% Formic acid

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

HPLC Method with MS detection

Column: Syncronis Aq column 150mm x 2.1mm

Mobile phase: gradient profile with A (Water – Ammonium Acetate 10mM) and B (Acetonitrile – Ammonium Acetate 1mM)

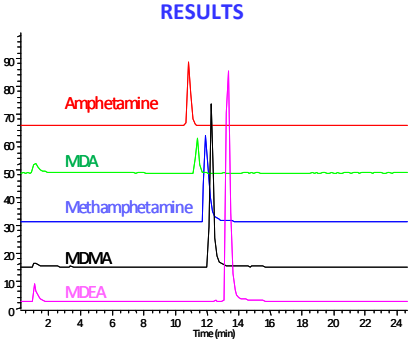
Time (min)	% A	% B
0	95	5
2	95	5
12	60	40
12.1	95	5

flow rate: 0.4mL/min

MS detection (ESI^+) : m/z 136 (Amphetamine) ; 180 (MDA); 150 (Methamphetamine); 194 (MDMA); 208 (MDEA)

Injection volume: 20µL.

RESULTS



Mass Chromatogram (SIM) obtained after AFFINIMIP® SPE Amphetamines clean-up of a human serum sample spiked at 100ng/mL with Amphetamine and its derivatives.

Recovery of Amphetamines in human serum spiked at 100ng/mL after AFFINIMIP® SPE Amphetamines clean-up and relative standard deviation calculated from results generated under reproducibility conditions (n=4).

Sample	Mean ng/mL	Recovery %	% RSD_R
Amphetamine	87.9	87.9	5.0
MDA	94.4	94.4	3.7
Methamphetamine	90.7	90.7	2.2
MDMA	106.2	106.2	2.5
MDEA	111.0	111.0	4.9

Catalog number: DG102-02

TETRACYCLINES, THEIR EPIMERS AND DOXYCYCLINE IN MILK AND SALMON

PROTOCOL OF CLEANUP

Sample preparation for Milk

Mix 1.5mL of Milk with 6mL of EDTA/Mc Ilvaine's Buffer and centrifuge at 4000rpm for 10 min at a temperature below 15°C. Collect the supernatant and add 750µL 1N NaOH solution. Adjust to pH 10 with a NaOH solution (this mixture was the loading solution).

Sample Preparation for Salmon based on AOAC 995.09 method

Blend 10g Salmon with 40mL of EDTA/Mc Ilvaine's Buffer during 30 s and stir during 10min with a magnetic stirrer. Centrifuge the mixture at 2500g for 10 min at a temperature < 15°C. Collect the supernatant Repeat this operation with 40mL buffer and again with 20mL of buffer. Then, gather all the supernatants and centrifuge during 20min at 2500g, filter on Buchner. Add 750µL 1N NaOH solution to the filtrate and adjust to pH 10 with a NaOH solution (this mixture was the loading solution).

Cleanup with a 1mL/10mg AFFINIMIP® SPE Tetracyclines cartridge

Equilibration
- 1mL Acetonitrile
- 1mL Water

Loading
- Loading solution (7.5mL)

Washing of interferences
- 1mL Water
- 2mL Water/Acetonitrile (60/40)

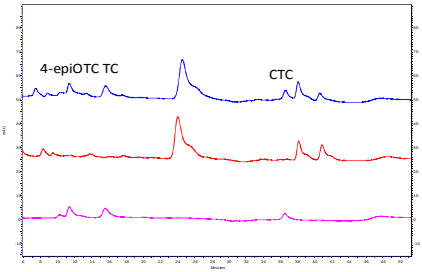
Drying 3 minutes

Elution (E)
- 2mL Methanol with 2% Formic acid

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

HPLC Method with UV detection same as p 72

RESULTS



UV Chromatograms (355nm) obtained after clean-up with AFFINIMIP® SPE Tetracyclines of 1.5mL of Milk spiked with Tetracycline, Chlortetracycline and 4-epioxytetracycline (4-epiOTC) at 50µg/L (blue) or not spiked (red) or of 1.5mL of water spiked with Tetracycline, Chlortetracycline and 4-epioxytetracycline at 50µg/L (pink)

Recovery of Tetracyclines after AFFINIMIP® SPE Tetracyclines clean-up of Salmon or milk spiked at 50 or 100µg/L and relative standard deviation calculated from results generated under **repeatability conditions** (n=3).

Molecules	Mean (µg/L)	Milk		Salmon
		R %	% RSDr	R%
Tetracycline	49.6	99.4	4.9	113
OTC	45.6	91.3	7.1	-
CTC	37.2	74.4	6.3	74
4-epiTC	47.9	95.9	5.1	-
4-epiCTC	108.4	108.4	15.0	97
4-epiOTC	43.7	87.4	9.1	71
DOX	43.8	88.0	2.9	89

Catalog number: FS112-02A

TETRACYCLINES, THEIR EPIMERS IN MEAT

PROTOCOL OF CLEANUP

Preparation of loading solution for Meat based on AOAC 995.09 method

10g meat were blend during 30 seconds with 40mL of EDTA/Mc Ilvaine's Buffer and stirred during 10min with a magnetic stirrer. The mixture was centrifuged at 2500g for 10 minutes at a temperature below 15°C. The supernatant was collected. This operation was repeated with 40mL of buffer and again with 20mL of buffer. Then, all the supernatants were gathered and centrifuged during 20min at 2500g, filtered on Buchner. 750µL 1N NaOH solution were added to the filtrate and adjusted to pH 10 with a NaOH solution (this mixture was the loading solution).

Cleanup with a 1mL/10mg AFFINIMIP® SPE Tetracyclines cartridge

Equilibration
1mL Acetonitrile
1mL Water

Loading
5mL Loading solution

Washing of interferences
1mL Water
2mL Water/Acetonitrile (60/40)

Drying 1 minute (only if elution is evaporated)

Elution (E)
2mL Methanol with 2% Formic acid

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

HPLC Method with UV detection

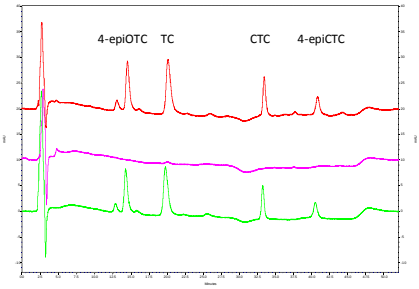
Column: Hypersil Gold C18 column 150mm x 2.1mm, 3µm

Mobile phase: gradient profile

Time min	% 10mM Oxalic Acid Water	% 10mM Oxalic Acid ACN	% MeOH
0	90	5	5
20	90	5	5
21	80	10	10
40	80	10	10
41	90	5	5

Flow rate: 0.2mL/min
UV detection: 355nm
Injection volume: 100µL.

RESULTS



UV Chromatograms (355nm) obtained after clean-up with AFFINIMIP® SPE Tetracyclines of meat spiked with Tetracycline, Chlortetracycline, 4-epichlortetracycline (4-epiCTC) and 4-epioxytetracycline (4-epiOTC) at 50µg/L (red), not spiked (pink) or of water spiked (green)

Recovery of Tetracyclines after AFFINIMIP® SPE Tetracyclines clean-up of Meat spiked at 200µg/kg (4-epiCTC at 400µg/kg)

Molecules	R% (n=2)
Tetracycline	98
Chlortetracycline	70
4-epichlortetracycline	74
4-epioxytetracycline	91

Catalog number: FS112-02A

TETRACYCLINES IN PORK KIDNEY TISSUS

PROTOCOL OF CLEANUP

Preparation of loading solution for pork kidney tissue based on AOAC 995.09 method

10g meat were blend during 30 seconds with 40mL of EDTA/Mc Ilvaine's Buffer and stirred during 10min with a magnetic stirrer. The mixture was centrifuged at 2500g for 10 minutes at a temperature below 15°C. The supernatant was collected. This operation was repeated with 40mL of buffer and again with 20mL of buffer. Then, all the supernatants were gathered and centrifuged during 20min at 2500g, filtered on Buchner. Around 10mL 1N NaOH solution were added to the filtrate and adjusted to pH **6.5** with a NaOH solution (this mixture was the loading solution).

Cleanup with a 1mL/10mg AFFINIMIP® SPE Tetracyclines cartridge

Equilibration

1mL Acetonitrile

1mL Water

Loading

4 to 5mL Loading solution

Washing of interferences

Wash the cartridge with 1mL of NaHCO3 1% in water

Immediately wash the cartridge with 2mL of deionized Water/Acetonitrile (60/40, v/v)

Drying 1 minute (only if elution is evaporated)

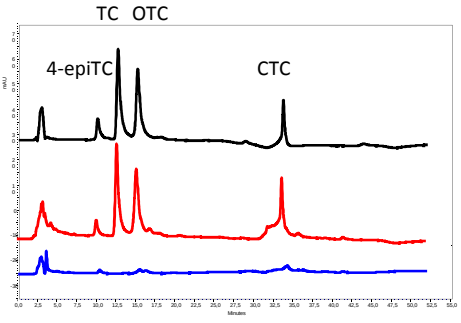
Elution (E)

2mL Methanol with 2% Formic acid

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

HPLC Method with UV detection same as p 72

RESULTS



UV Chromatograms (355nm) obtained after clean-up with AFFINIMIP® SPE Tetracyclines pork kidney tissue (red) or water (black) spiked with 910µg/kg Tetracycline, 980µg/kg Oxytetracycline and 860µg/kg Chlortetracycline as well as pork kidney not spiked (blue)

Recovery and repetability of Tetracyclines after AFFINIMIP® SPE Tetracyclines clean-up of Pork kidney tissue at 910µg/kg for TC; 980µg/kg for OTC and 860µg/kg for CTC

Molecules	R% (n=5)	RSDr (%)
Tetracycline (TC)	85	5,4
Chlortetracycline (OTC)	79	2,8
Oxytetracycline (CTC)	80	4,2

Catalog number: FS112-02A

ANTIBACTERIAL AMINOGLYCOSIDES ON MILK OR MEAT

streptomycin (STR) Dihydrostreptomycin (DHS)
hygromycin B (HB)
kanamycin (KM)
apramycin (APM)
destomycin A (DA)
amikacin (AK)
Paromomycin (PM)
Tobramycin

PROTOCOL OF PURIFICATION FOR MEAT and MILK

Sample preparation for meat

2g meat are mixed during 10min with 10mL extraction buffer (10 mM KH2PO4, 0.4 mm EDTA, 2% trichloroacetic acid). Then centrifuge during 10 min at 4 000 rpm, and collect the supernatant.

Repeat two times. Adjust the pH 7.5 ~ 8.0 with 5 M NaOH (0.3 ~ 0.4 mL) to obtain the loading solution.

Sample preparation for Milk

Mix 5mL milk in 300µL 50% trichloroacetic acid during 10min. Then centrifuge during 15min at 5000rpm and collect the supernatant. Add 300µL 50% trichloroacetic acid to the supernatant and centrifuge again 15sec.

Adjust the pH 7.5 ~ 8.0 with 1M NaOH to obtain the loading solution.

Purification with a 6mL/200mg AttractSPE™ HLB cartridge

Equilibration
5mL Methanol
5mL Water

Loading
Loading solution

Washing
5mL water

Drying 5 min

Elution (E)

2x 3mL 100mM Heptafluorobutyric acid (HFBA) in Acetonitrile –Methanol (2+1, v/v)
Evaporate under nitroger at 50°C
Reconstitute with 1mL 20mM HFBA solution before analysis.
Detection LC-MS/MS

Catalog number: HLB-50.S.6.200

MULTI-RESIDUE DETERMINATION OF SEVERAL FAMILIES OF ANTIBIOTICS IN KIDNEY

PROTOCOL OF PURIFICATION

Sample preparation: Vortex 1 g of kidney with 10mL of McIlvaine/EDTA buffer during 1min. Shake for 15min and ultrasonic for 5 min. Centrifuge at 3800g for 10min at 5°C. Filter and collect supernatant.
Repeat extraction with 3mL buffer solution.
Combine the supernatants to obtain the loading solution.

Purification with a 6mL/200mg AttractSPE™ HLB cartridge

Equilibration
5mL Methanol
5mL Water

Loading
Loading solution

Washing
6mL water/methanol (95/5, v /v)

Drying 10 min

Elution (E)
6mL Methanol

Evaporate under nitroger at 40°C and reconstituted with 1mL water/acetonitrile (90/10, v/v). Vortex, centrifuge and filter
Analysis: LC-MS/MS

73 substances measured from drug families:
Quinolones
Macrolides
Lincosamides
Sulfonamides
Penicillins
Cephalosporine
Pleuromutilins
Diamino pyrimidine derivatives

Catalog number: HLB-50.S.6.200

MULTI-RESIDUE DETERMINATION OF NSAID IN MUSCLE TISSUE

PROTOCOL OF PURIFICATION

Sample preparation: Shake 2g of homogenized meat with 10mL ACN for 2min. Centrifuge during 5min at 5500rpm and evaporate the supernatant at 60°C under nitrogen. Reconstitute with 100µL Methanol–900µL Water to obtain the loading solution.

Purification with a 3mL/60mg AttractSPE™ HLB cartridge

Equilibration
1mL Methanol
1mL Water

Loading
All the loading solution

Washing of interferences
1mL Methanol/Water (5/95)

Drying under vaccuum

Elution (E)
1mL Methanol
1mL Hexane with 10% Acetic acid

The elution fraction was then evaporated at 60°C under nitrogen and reconstituted before HPLC analysis.
Analyse: LC/MS-MS

Regulations - MRL for NSAID in bovive muscle:
Carprofen 500µg/kg
Flunixin 20µg/kg
Tolfenamic acid 50µg/kg
Meloxicam 20µg/kg

Salicylic acid
Phenylbutazone
Flunixin
Tolfenamic acid
Meloxicam
Desoximethasome (IS)
Ketoprofen

Catalog number: HLB-50.S.3.60

PENICILLIN BASED ANTIBACTERIALS ON MUSCLES

PROTOCOL OF PURIFICATION FOR MUSCLES

Sample preparation for muscle:

2g muscle samples are mixed in a 50mL centrifuge tube with 10mL 0.1 M sodium phosphate buffer (pH 4.5) and then homogenized. Add 2.5 mL 0.17 M sulfuric acid, 2.5 mL 5% sodium tungstate and mix it well,. Centrifuge at 5 000rpm during 15 min.

The supernatant was adjusted at pH 8.1 ~ 8.5 with 5M NaOH, centrifuged at 5 000rpm during 15 min and the supernatant is collected and mix with 10mL NaCl (20%) to obtain the loading solution.

Purification with a 6mL/200mg AttractSPE™ HLB cartridge

Equilibration
- 5mL Methanol
- 5mL Water
- 5mL 2% NaCl

Loading
- Loading solution

Washing
- 5mL 2% NaCl
- 5mL 25mM PBS (pH 9,0)

Drying 5 min

Elution (E)

2x 3mL Acetonitrile

Evaporate under nitroger at 40°C and reconstituted before analysis.

Detection LC-MS/MS

Ampicillin,
Amoxicillin
Penicillin G or benzypenicillin
Penicillin V
Oxacillin
Nafcillin,
Cloxacillin
Dicloxacillin

Catalog number: HLB-50.S.6.200

AttractSPE™ HLB

ERYTHROMYCIN AND CLINDAMYCIN

PROTOCOL OF PURIFICATION

Sample preparation

Mix 2g of crushed samples with 6mL of 2% acetic acid solution in centrifuge tube and then centrifuge at 1200g for 10 min to form the loading solution.

Purification with a 6mL/200mg AttractSPE™ HLB cartridge

Equilibration
- 5mL Methanol
- 5mL Water

Loading
- Loading solution

Washing
- 5mL distilled water

Elution (E)

6mL Methanol

Detection LC-MS

Regulation
EC 37/2010
Erythromycin
200kg/kg muscle

Catalog number: HLB-50.S.6.200

QUINOXALINE-2 -CARBOXYLIC ACID AND 3-METHYL QUINOXALINE-2-CARBOXYLIC ACID IN MUSCLE, LIVER, KIDNEYS

PROTOCOL OF PURIFICATION

Sample preparation

Step 1: Mix 5 g homogenized sample with 100µL of internal standard (Quinoxaline-2-carboxylic acid-D4) and 10mL solution of 10% Methanol in water containing 5% metaphosphoric acid in a 50 mL centrifuge tube and shake, then centrifuge at 4500rpm for 10 min at 30°C.

Step 2: Collect the supernatant in a 50mL centrifuge tube. Repeat the step 1 with 10mL solution of 10% Methanol in water containing 5% metaphosphoric acid and centrifuge at 5000rpm for 20 min at 30°C.

Sept 3: Combine the supernatants (~20mL)

Step 4: Mix vigorously with 10mL Ethyl Acetate for 15min then centrifuge at 5000rpm for 10 min at 30°C and collect the supernatant.

Step 5: repeat step 4, combine the supernatants, concentrate them at 60°C under nitrogen. The residue is dissolved in 5mL HCl 0.1M.

Purification with a 3mL/60mg AttractSPE™ SAX cartridge

Equilibration
- 3mL Methanol
- 3mL Water

Loading
- Loading solution

Washing
- 3mL water

Drying for 5min

Elution (E)
- 3mL Methanol - 0.1M HCl (90-10 v/v)

This eluate was dried at 60 ℃ under nitrogen

Analysis: LC-MS/MS

Catalog number: SAX-50.S.3.60

MULTI-CLASS METHOD OF ANTIBIOTICS IN MILK

PROTOCOL OF PURIFICATION

Sample preparation: Mix 1mL Milk and 1mL Acetonitrile in a PP centrifuge tube. Voxtex for 10-15s and centrifuge at 4000rpm (4°C) for 10min. Collect the supernatant (avoiding any visible fat layer) and add to a glass test tube containing 9mL 0,1% Formic acid.
Vortex the tube during 10 s to obtain the loading solution.

Purification with a 3mL/60mg AttractSPE™ HLB cartridge

Equilibration
3mL Acetonitrile
3mL water with 0,1% formic acid/Acetonitrile (95/5, v/v)

Loading
Loading solution

Washing
2x2mL 0,1% formic acid

Drying 30s

Elution (E)
2.5mL Acetonitrile/Methanol 70/30
Analysis: LC-MS/MS

25 substances measured from
Fluoroquinolones
Beta lactam
Sulfonamide
Macrolides

Catalog number: HLB-50.S.3.60

Same method as FDA Lab information bulletin LIB# 4443, Susan B. Clark, Joseph M. Storey, Sherri B. Turnipseed

VANCOMYCIN IN FISH

AttractSPE™ SCX

PROTOCOL OF PURIFICATION

Sample preparation:
Step 1: Mix 5 g homogenized sample with a 15mL solution of 20% ACN in water in a 50 mL centrifuge tube and shake for 20 min, then centrifuge at 7 600rpm for 10 min.
Step 2: Collect the supernatant in a 50mL centrifuge tube. Repeat the step 1 with 10mL solution of 20% ACN in water.
Combine the supernatants (~25mL) and mix vigorously with 10mL Hexane for 10min then centrifuge at 7 600rpm for 10 min and remove Hexane to obtain the loading solution.

Purification with a 3mL/60mg AttractSPE™ SCX cartridge

Equilibration
3mL Methanol
3mL Water- 0.1% Formic acid

Loading
3mL Loading solution

Washing
3mL water

Elution (E)
3mL Methanol with 3% Ammonium hydroxide

This eluate was dried at 50 °C under nitrogen and reconstituted in 1mL water and filtered at 0.2µm.
Analysis: LC-MS/MS

Catalog number: SCX-50.S.3.60

GLUCOCORTICOIDS

PROTOCOL OF PURIFICATION

Sample preparation

Mix 2 g of the sample and 10mL of acetate buffer solution (3M, pH 4.6) in a 50 mL centrifuge tubes and homogenize for about 2 minutes. Make an enzymatic hydrolysis by adding 50µL Helix pomatia β-Glucuronidase/Arylsulfatase for 1h in an oven at 60 °C . After cooling at RT, add 8 mL CAN and centrifuge at 4500rpm for 10 min. Repeat the above steps. Collect the supernatants and concentrate under nitrogen at 50 °C. Dissolve the residue in 1mL Ethanol and add 5mL of distilled water to obtain the loading solution.

Purification with a 6mL/500mg AttractSPE™ HLB cartridge

Equilibration
- 5mL Methanol
- 5mL Water

Loading
- Loading solution

Washing
- 5mL acetone/distilled water (2/8, v / v)
- 5mL n-hexane

Drying 2 min

Elution (E)

6mL Ethyl Acetate

Evaporate under nitrogen at 50°C and reconstituted before analysis with 1mL mobile phase. Centrifuge at -4 °C, 15000rpm during 15 min. The clear supernatant is filterer 0.2 µm nylon filter and analyzed by LC-MS/MS after the filtration

Detection LC-MS/MS

Cortisone, Corticosterone, Aldosterone, Betamethasone, Dexamethasone, Flumethasone, Prednisone, Prednisolone, Methylprednisolone

Catalog number: HLB-50.S.6.500

SULFONAMIDES IN MILK

PROTOCOL OF PURIFICATION

Purification with a 3mL/60mg AttractSPE™ SCX cartridge

Equilibration
- 2mL Methanol
- 2mL Water

Loading
- 5mL Milk

Washing of interferences
- 2mL Methanol/Water (5/95)
- 1mL 0.5M HCl
- 2mL Methanol/Water (20/80)

Elution (E)
- 2.5mL Ammonium bicarbonate/Methanol (10/90)

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

Analyse LC/MS-MS

Regulations for Sulfonamides:
Sulfadimethoxine 0.01ppm in milk (U.S. FDA 21 CFR 556.640)
Sulfaethoxypyridazine 0 in milk (U.S. FDA 21 CFR 556.650)
EC 37/2010 100µg/kg Milk

Catalog number: SCX-25.S.3.60

MULTI-CLASS METHOD OF ANTIBIOTICS IN DISTILLER GRAINS

PROTOCOL OF PURIFICATION

Sample preparation

Mix 5mL Distillers grains with 20mL 1,5mM EDTA and 20mL 1% Trichloroacetic acid in water in a 50mL PP centrifuge tube. Shake for 15min and centrifuge at 4000rpm for 10min. Collect the supernatant and dilute it with 150mL water.

Repeat extraction with 30mL methanol and centrifuge and combine supernatants.

Dilute the supernatants to 200mL with water to obtain the loading solution.

Purification with a 6mL/150mg AttractSPE™ HLB cartridge

Equilibration

3mL Methanol

3mL water with Trichloroacetic acid (pH~4)

Loading

10mL Loading solution

Drying under vacuum for 5min

Washing

5mL water

Drying under vacuum for 5min

Elution (E)

2.5mL Methanol

Evaporate eluate to about 1mL under nitrogen at 35°C.

Analysis: LC-MS/MS

Analyses of 13 antibiotics Ampicillin, bacitracin A, erythromycin, tylosin, clarithromycin, penicillin G, virginiamycin M1 and monensin

Catalog number: HLB-50.S.6.150

Same method as FDA Lab information bulletin LIB# 4438 David N. Heller G.K. Hemakanthi de Alwis

AttractSPE™ HLB

PRAZIQUANTEL AND TIAMULIN

PROTOCOL OF PURIFICATION

Sample preparation

Mix 2g of pulverized samples with 6mL of 2% ammonium hydroxide solution in centrifuge tube and then centrifuge at 1200g for 10 min to form the loading solution.

Purification with a 6mL/200mg AttractSPE™ HLB cartridge

Equilibration

5mL Methanol

5mL Water

Loading

Loading solution

Washing

5mL distilled water

Elution (E)

6mL Methanol

Analysis: LC-MS

Catalog number: HLB-50.S.6.200

CEPHALEXIN IN FISH

PROTOCOL OF PURIFICATION

Sample preparation

Mix 2g of homogenized samples with 5mL of 10% trichloroacetic acid solution in a 50mL centrifuge tube and then centrifuge at 3000g for 10 min. Collect the supernatant and make a 1:1 mix with a 4% phosphoric acid to form the loading solution.

Purification with a 3mL/60mg AttractSPE™ SCX cartridge

Equilibration

2mL Methanol

2mL Water

Loading

Loading solution

Washing

2mL 2% Formic acid

2mL Methanol

Elution (E)

4mL Methanol with 5% Ammonium hydroxide

This eluate was concentrated at 50 ℃ under nitrogen and reconstituted with 200µL 0.1% formic acid containing 10% acetonitrile. The solution was centrifuged at 12 000 G for 10 minutes and the supernatant was filtered with a membrane filter.

Analysis: LC-UV (260nm)

Catalog number: SCX-50.S.3.60

VALNEMULIN AND TIAMULIN IN FISH

PROTOCOL OF PURIFICATION

Sample preparation

Step 1: Mix 1 g homogenized sample with a 10mL solution of 40-60 (v:v) ACN-0,01M HCl in a 50 mL centrifuge tube and shake at 300rpm for 15 min, then centrifuge at 10 000rpm for 10 min.

Step 2: Collect the supernatant in a 50mL centrifuge tube. Repeat the step 1 with the lower layer of the first centrifuge tube.

Combine the supernatants (~20mL) and mix vigorously with 20mL Hexane and remove Hexane to obtain the loading solution.

Purification with a 3mL/60mg AttractSPE™ SCX cartridge

Equilibration

3mL Methanol

3mL Water

Loading

3mL Loading solution

Washing

3mL 40-60 (v:v) ACN-0,01M HCl

Drying 1min

Elution (E)

3mL Methanol with 5% Ammonium hydroxide

This eluate was dried at 40 ℃ under nitrogen and reconstituted in the mobile phase.

B. Assay conditions

Analysis: LC-MS/MS

Catalog number: SCX-50.S.3.60

PHARMACEUTICALS AND PERSONAL CARE PRODUCTS (GROUP 1 COMPOUNDS) OF EPA METHOD 1694

PROTOCOL OF PURIFICATION

Sample preparation

Filtrate 1L solution and adjust the pH to 2 while stirring the water. Add 500mg Na_4 EDTA and mix. Equilibrate during 1-2h to obtain the loading solution.

Purification with a 6mL/200mg AttractSPE™ HLB cartridge

Equilibration
- 20mL Methanol
- 6mL Water
- 6mL Water pH 2

Loading

1L of loading solution, pH 2

Washing

10mL water

Drying 5 min

Elution (E)

6mL Methanol or 6mL Methanol – Acetonitrile (50/50)

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

EPA methods 1694

Acetaminophen	Ormetoprim
Caffeine	Sarafloxacin
Carbadox	Sulfachloropyridazine
Cefotaxime	Sulfadiazine
Ciprofloxacin	Sulfamerazine
Clinafloxacin	Sulfamethazine
Codeine	Sulfamethizole
Cotinine	Sulfamethoxazole
1,7-Dimethylxanthine	Sulfanilamide
Enrofloxacin	Sulfathiazole
Lincomycin	Thiabendazole
Lomefloxacin	Trimethoprim
Norfloxacin	
Ofloxacin	

Catalog number: HLB-50.S.6.200

AttractSPE™ HLB

MISCELLANEOUS DRUGS IN WASTEWATER

PROTOCOL OF PURIFICATION

Sample preparation

Filtrate 500mL to 1L of wastewater with 0,45µm glass fiber to form the loading solution.

Purification with a 3mL/60mg AttractSPE™ HLB cartridge

Equilibration
- 3mL Ethyl Acetate
- 3mL Methanol
- 3mL Water

Loading

Loading solution (15mL/min)

Washing

3mL Methanol/ water (5/95, v/v)

3mL n-hexane

Elution (E)

3x1mL Ethyl Acetate

Evaporate under nitrogen and reconstituted with 0.5mL Methanol.

Analysis: LC-DAD-Fluorescence

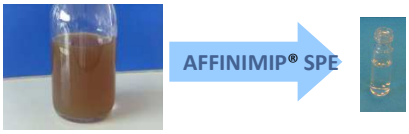
- Caffeine
- Acetaminophen
- Diclofenac
- Ibuprofen
- Ketoprofen
- Naproxen
- Carbamazepine

Catalog number: HLB-50.S.3.60

Analysis of PESTICIDES

AMINOPYRALID, CLOPYRALID AND PICLORAM IN COMPOST AND WATER

Efficient clean-up and enrichment



Loading solution

Elution solution

PROTOCOL OF PURIFICATION

Sample preparation for compost

5g of compost sample and 100mL water are shaked during 60minutes. Centrifuge at 3000g for 10min and then filter the solution with a 4-7µm filter. This solution is used as the loading solution.

Purification with a 3mL/60mg AFFINIMIP® SPE Picolinic Herbicides cartridge

Equilibration
- 2mL Acetonitrile
- 1mL Water

Loading
- 3mL of loading solution

Washing of interferences (W1)
- 1mL Water

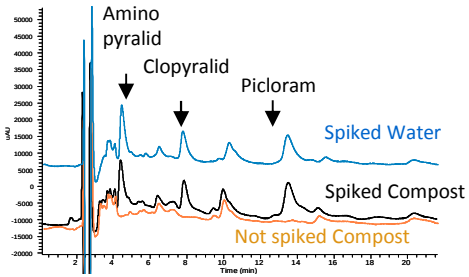
Drying by applying vacuum 1 min

Washing of interferences (W2)
- 1mL Acetonitrile

Elution (E)
- 3mL 98/2 Ethyl acetate / Trifluoroacetic acid

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

RESULTS



UV chromatogram of compost or water spiked with Aminopyralid, Picloram and Clopyralid after AFFINIMIP®SPE Picolinic Herbicides clean-up

Recovery and repeatability of Picloram, Aminopyralid and Clopyralid in compost (n=3) and after AFFINIMIP® SPE Picolinic Herbicides Clean-up.

Analytes	Recoveries % for Water	Recoveries % for Compost	% RSDr compost
Aminopyralid	95	84	3
Clopyralid	109	120	4
Picloram	88	89	3

Catalog number: FS115-02

GLYPHOSATE AND AMPA IN WATER

Efficient clean-up and enrichment

Also tested with up to 20% Methanol for percolation and washing

PROTOCOL OF PURIFICATION

Purification with a 3mL AFFINIMIP® SPE Glyphosate – AMPA cartridge

Equilibration
- 6mL Water

Loading
- Water

Washing of interferences (W1)
- 3mL Water

Elution (E)
- 3mL HCl solution (100mM)

The elution fraction was then derivatized with FMOC and dissolved in the mobile phase before HPLC analysis.

Method UPLC – MS/MS
Column: Acquity UPLC HSS T3 (2.1mm x 100mm, 1,8µm)
Mobile phase:

Time (min)	% Water/Ammonium Acetate 5mM	% ACN
0	90	10
2	90	10
7	50	50
7.5	0	100
11	0	100

flow rate: 0.2mL/min
MS detection: m/z 321 (ESI^-)
Injection volume: 20µL.

RESULTS

Recovery Yields of AMPA and Glyphosate in water for a range of concentration of 50 to 450ng/L of Glyphosate and of 50 to 550ng/L of AMPA after AFFINIMIP® SPE Glyphosate - AMPA clean-up

Sample	Recoveries %
GLYPHOSATE	>80%
AMPA	>75%

Poster:
Extraction SPE basée sur un polymère à empreintes moléculaires pour l'extraction du glyphosate et de son métabolite (AMPA) dans des eaux souterraines, by BRGM and ICOA (ANR project Origami), AFSEP 2014 Paris.

New Selective SPE Clean-Up Method Based on Molecularly Imprinted Polymers for Glyphosate and AMPA Analysis with and without Derivatization for Water, Food and Feed, Pittcon 2015 New Orleans.

Catalog number: FS113-02

GLYPHOSATE AND AMPA IN WATER

MIP performance not affected by physico chemical properties of Water

Physico chemical properties of tested waters

Salt concentrations (mg/L) and pH of analyzed solutions

	Ca	Na	Mg	K	HCO3	Cl	NO3	SO4	Fe	pH
Groundwater	15,7	11,3	4,9	1,3	76	9,7	<0,5	1,2	7,5	7,1
Groundwater	22,3	105,7	17	4,7	136	159	8,9	15,8	0,17	6,4
Groundwater	104,1	13,9	6,9	1,8	203	28,1	113,7	33		7,1
Geothermal water	799	5163,5	189,5	71,9		9759,7		702,2	3,2	
Mineral water	80	6,5	24	1	360	3,8	3,7	12,6		7,2

MIP performance for tested waters

Above five waters spiked at various concentrations with AMPA and Glyphosate

Sample	Concentration range	Recoveries %
Glyphosate	50 to 450ng/L	>80%
AMPA	50 to 550ng/L	>75%

Method UPLC – MS/MS

Column: Acquity UPLC HSS T3 (2.1mm x 100mm, 1,8µm)

Mobile phase:

Time (min)	% Water/Ammonium Acetate 5mM	% ACN
0	90	10
2	90	10
7	50	50
7.5	0	100
11	0	100

flow rate: 0.2mL/min

MS detection: m/z 321 (ESI^-)

Injection volume: 20µL.

Acknowledgment: French ANR project ORIGAMI (ANR ECOTECH 2011 ORIGAMI 11 ECOT 003)

Catalog number: FS113-02

16 PESTICIDES FROM GROUNDWATER

PROTOCOL OF PURIFICATION

Purification with a 6mL/200mg AttractSPE™ HLB cartridge

Equilibration
- 10mL Dichloromethane
- 10mL Acetonitrile
- 10mL Water

Loading

1L Water sample

Washing of interferences
- 5mL Water/Methanol 95/5

Elution (E)

5mL Acetonitrile
5mL Methanol

Analyse HPLC

Desysopropylatrazine, Desethylatrazine, Aldocarb, Simazine, Carbofuran, Metalaxyl, Atrazine, 2, 4-D, Metazachlor, Dicloran, Phenmedipham, Linuron, Iprodione, Procymidone, Fenitrothion, Vinclozolin

Catalog number: HLB-50.S.6.200

AttractSPE™ HLB

PESTICIDES FROM SURFACE WATER

PROTOCOL OF PURIFICATION

Purification with a 6mL/200mg AttractSPE™ HLB cartridge

Equilibration
- 3mL Methanol/Acetonitrile 50/50
- 6mL Water

Loading

1L Water sample (+0.5g NaCl)

Drying

Elution (E)

3mL Acetonitrile/Methanol 50/50

Analyse HPLC

Desethylsimazin, 2, 6-Dichlorbenzamid, Ethidimuron, Chloridazon, Desethylatrazin, Desethylsebuthylazin, Bromacil, Simazin, Metribuzin, Desethylterbuthylazin, Metabenzthiazuron, Chlortoluron, Atrazin, Diuron, Isoproturon, Matazaclor, Terbumeton, Sebuthylazin, Propazin, Dimefuron, Terbuthylazin, Triadimenol, Epoxiconazol, Terbutryn, Metolachlor, propiconazol, Kresoxim- methyl

Catalog number: HLB-50.S.6.200

ACETAMIDE HERBICIDES IN DRINKING WATER

PROTOCOL OF PURIFICATION
Purification with a 3mL/60mg AttractSPE™ HLB cartridge

Equilibration
- 3mL Methanol
- 2mL Water

Loading
150mL Water sample

Washing of interferences
- 1mL Water

Elution (E)
1mL Methanol

Analyse HPLC

Metolachlor metabolite, 2-Chloro 2, 6 diethylacetanilide, 2,6 Diethylaniline, Alachlor, Metolachlor

Catalog number: HLB-50.S.3.60

AttractSPE™ HLB

HERBICIDES IN DRINKING WATER

PROTOCOL OF PURIFICATION
Purification with a 3mL/60mg AttractSPE™ HLB cartridge

Equilibration
- 3mL Methanol
- 6mL Water

Loading
75mL Water sample

Washing of interferences
- 1mL Water

Elution (E)
1mL Methanol

Analyse HPLC

Desispropylatrazine, Hydroxyatrazine, Desethylatrazine, Simazine, Cyanazine, Atrazine

Catalog number: HLB-50.S.3.60

Analyses of
OTHER RESIDUES AND MISCELLANEOUS

NNAL IN URINE

PROTOCOL OF CLEANUP

Cleanup with a AFFINIMIP® SPE NNAL cartridge

Equilibration
- 2mL Toluene
- 2mL 10% MeOH-CH2Cl2
- 3mL CH2Cl2
- Dry
- 1mL CH2Cl2
- 1mL MeOH
- 1mL Water

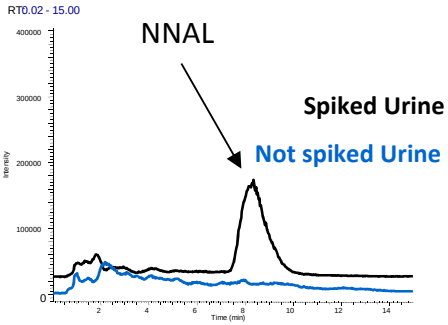
Loading
- 2mLUrine or Water

Washing of interferences
- 2mL Water
- Dry 10min
- 1mL Toluene
- 1mL Toluene : CH2Cl2 9:1
- 1mL Toluene : CH2Cl2 4:1
- Dry 2min

Elution of phenolic compounds

2mL 10% MeOH-CH2Cl2

Recovery for urine 112%



LC-MS chromatogram of urine spiked with NNAL (spiked at 100ng/mL) after AFFINIMIP®SPE NNAL clean-up

HPLC Method with LC-MS/MS detection

Column: Syncronis aQ column 150mm x 2.1mm
Mobile phase: Water – 0.1% Formic Acid
flow rate: 0.2mL/min
MS detection: m/z 322 (ESI^+)
Injection volume: 20µL.

Catalog number: DG103-02

MELAMINE IN FOOD

PROTOCOL OF PURIFICATION

Sample preparation: Add 5mL water and 5mL Acetonitrile to 1g pulverized sample. Shake thoroughly for 30 min and centrifuge for 10minutes at 2600 rpm.
The supernatant is filtered through 0,45µm membrane filter to obtain the loading solution.

Purification with a 6mL/150mg AttractSPE™ SCX cartridge

Equilibration
- 5mL Acetonitrile
- 5mL 4% Formic acid in Water

Loading

3mL 4% Formic acid in Water
2mL of loading solution

Washing of interferences
- 5mL Acetonitrile
- 5mL 0.2% diethyamine in Acetonitrile

Elution (E)

4mL 2% diethylamine in Acetonitrile

The elution fraction was filtered and then evaporated under nitrogen at 50°C and dissolved in the mobile phase before HPLC analysis.

Analyse LC-MS/MS

Catalog number: SCX-25.S.6.150

POLYCYCLIC AROMATIC HYDROCARBONS FROM DRINKING WATER

PROTOCOL OF PURIFICATION

Purification with a 6mL/200mg AttractSPE™ HLB cartridge

Equilibration
- 5mL Dichloromethane
- 5mL Methanol
- 5mL Water

Loading

500mL Water sample

Washing of interferences
- 6mL Water

Elution (E)

8mL Dichloromethane

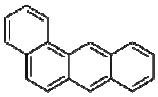
Analyse HPLC

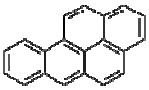
Naphtalene, Acenaphthylene, Acenaphthene, Fluorene, Phenanthrene, Anthracene, Fluoranthene, Pyrene, Benzo(a)anthracene, Chrysene, Benzo(b)fluoranthene, Benzo(k)fluoranthene, Benzo(a)pyrene, Dibenzo(g, h, l)perylene, Benzo(a)pyrene, Benzoperylene, Indenopyrene

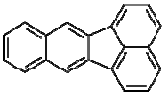
Catalog number: HLB-50.S.6.200

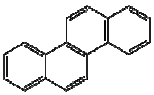
POLYCYCLIC AROMATIC HYDROCARBONS FROM OLIVE OIL

Regulations for B[a]P in oil and fats:
Europe (EC 208/2005) : 2µg/Kg


Benzo[a]anthracen
B[a]A


Benzo[a]pyren
B[a]P


Benzo[a]fluoranthen
B[a]F


Chrysen (CHR)

PROTOCOL OF CLEANUP

Sample preparation
Edible oil is diluted by 10 with cyclohexane. This solution is used as the loading solution.

Cleanup with a 3mL AFFINIMIP® SPE PAHs cartridge

Loading
- 1mL of loading solution

Washing of interferences (W1)
- 3mL Cyclohexane

Elution (E)
- 3mL Ethyl acetate

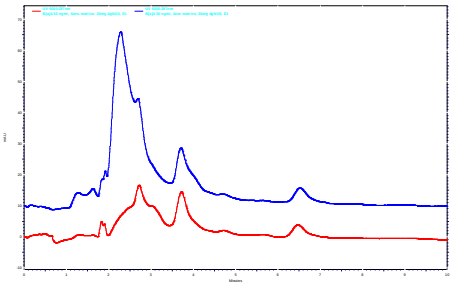
The elution fraction was then evaporated and dissolved in the mobile phase before HPLC analysis.

RESULTS

Recoveries of PAHs in cyclohexane and Olive oil after **AFFINIMIP® SPE PAHs** Clean-up

PAHs	Yield cyclohexane	Yield Olive oil
B[a]A	101%	108%
B[a]P	83%	120%
B[b]F	91%	111%
CHR	91%	72%

RESULTS



UV chromatogram of B[a]A (30ng/mL) in cyclohexane (red) and olive oil (blue) after Clean-up by **AFFINIMIP® SPE PAHs**.

Catalog number: FS119-02

HYDROXYLATED POLYCYCLIC AROMATIC HYDROCARBONS FROM SOIL

PROTOCOL OF CLEANUP

1g of soil spiked with 0.2mg/kg of hydroxylated PAH was extracted by microwave assisted extraction (MAE) with 25mL acetonitrile at 120°C for 30min to form the loading solution.
Cleanup with a 3mL/100mg AFFINIMIP® SPE Phenolics cartridge

Equilibration
4mL Toluene
2x4mL Acetonitrile
Loading
25mL of loading solution
Washing of interferences
4mL Acetonitrile
Drying
Elution (E)
6mL Methanol – 2 Acetic acid

RESULTS

Recoveries of OH-PAHs after clean-up with AFFINIMIP® SPE Phenolics for a linear range of 0.002-0.050 µg/mL

	Mean Recoveries % (%, mean ± SD, n=5)	LOD (µg/g)	LOQ (µg/g)	RSD%
2-OHNaph	79 ± 5	0.003	0.010	7
2-OHFlu	93 ± 6	0.003	0.011	9
9-OHPhe	89 ± 2	0.007	0.023	2
1-OHPyr	68 ± 6	0.014	0.044	8

HPLC-Fluorescence Method
Column: Envirosep PP C18 , 150mm x 4.6mmx5µm
Mobile phase:

Time (min)	% Water	% ACN
0	55	45
3	55	45
10	0	100

Flow rate: 1mL/min
Injection volume: 20µL.

Publications
Data extracted from the article Molecularly imprinted polymers-liquid chromatography/fluorescence for the selective clean-up of hydroxylated polycyclic aromatic hydrocarbons in soils, O. Baltrons, M. Lopez-Mesas, C. Palet, F. Lederf and F. Portet-Koltalo, *Anal. Methods*, 5, 6297-6305 2013.

Catalog number: FS103-02

METANEFHRINES IN PLASMA COMPARISON WITH WCX CARTRIDGES

PROTOCOL OF CLEANUP

Sample preparation

The plasma or serum is diluted by 5 with water. This solution is used as the loading solution.

Cleanup with a 1mL AFFINIMIP® SPE Metanephrines cartridge

Equilibration

- 1mL of phosphate buffer pH 7
- 2mL Water

Loading

1.5mL of loading solution

Washing of interferences (W1)

- 1mL Water
- 500µL Water/Methanol (60/40)

Drying 10 seconds

Washing of interferences (W2)

- 500µL Methanol

Elution (E)

1mL Methanol – 5% Acetic acid

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

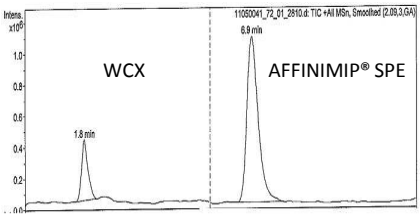
RESULTS

Recoveries of MN and NMN at a contamination level of 500nM in rabbit plasma after AFFINIMIP® SPE Metanephrines Clean-up and relative standard deviation calculated from results generated under reproducibility conditions (Analysis by LC-MS).

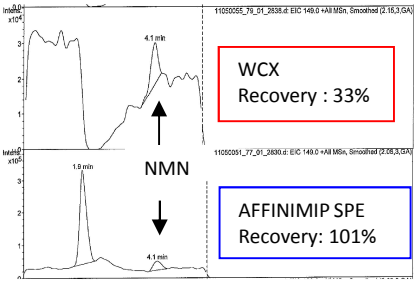
Analytes	Recovery %	% RSD_R
Metanephrine	79.4	6.3
Normetanephrine	109	11

Catalog number: DG101-02A

RESULTS



Analysis by LC-MS/MS: Total Ion Current of a calf serum after Cleanup by AFFINIMIP® SPE Metanephrines. The sample naturally contained Metanephrine. **Concentration of MN found: 30nM**. In parallel, a SPE was performed on a protocol developed for the analysis of MN using WCX cartridges: the concentration obtained was 7nM for the same sample.



Analysis by LC-MS/MS: Selected ion monitoring of Normetanephrine (m/z 180). Chromatograms obtained after Cleanup by AFFINIMIP® SPE Metanephrines or by WCX of a calf serum spiked at 27nM with Normetanephrine.

HPLC Method with LC-MS/MS detection

Column: Syncronis aQ column 150mm x 2.1mm

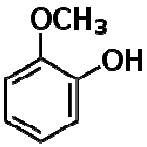
Mobile phase: Water – 0.1% Formic Acid

flow rate: 0.2mL/min

MS detection: m/z 322 (ESI^+)

Injection volume: 20µL.

GUAIACOL IN RED/WHITE WINE



General structure of Guaîacol

PROTOCOL OF CLEANUP

Cleanup with a 3mL/100mg AFFINIMIP® SPE Phenolics cartridge

Equilibration
- 3mL Acetonitrile
- 3mL Water

Loading
- Up to 2mL of red or white wine

Washing of interferences
- 3mL Water / Acetonitrile (80/20 v/v)

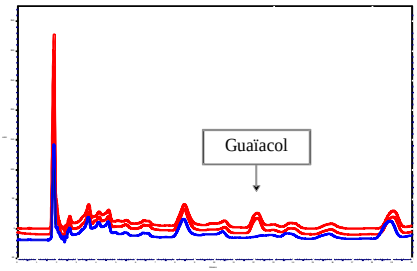
Elution (E)
- 2mL Methanol

Recovery yields and reproducibility evaluated with 3 cartridges and 3 different batches of AFFINIMIP® SPE Phenolics by matrix (n=9)

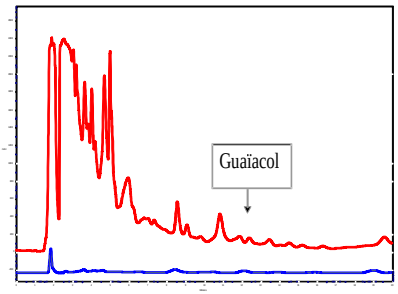
	C° (µM)	Rec. %	RSD_R %
Red wine 1	0.1	88.1	3.9
Red wine 2	0.1	93.1	3.7
White wine 1	0.02	96.8	1.7
White wine 2	0.02	93.5	2.6

Catalog number: FS103-02

RESULTS



Chromatograms obtained after clean-up with AFFINIMIP® SPE Phenolics of red wine spiked with Guaïacol (0.1µM) (red) or not spiked (blue).



Chromatograms obtained before (red) and after (blue) clean-up with **AFFINIMIP® SPE Phenolics** of red wine spiked with Guaïacol (0.1µM)

HPLC-UV Method
Column: Thermo Hypersil gold, 150mm x 4.6mm
Mobile phase: 15/85 (v/v) Acetonitrile Water
Flow rate: 1mL/min
Detection: UV - 272nm
Injection volume: 100µL.

CARNOSIC ACID IN MEAT

PROTOCOL OF CLEANUP

Sample preparation

25g of turkey was mixed with 200mL of 74.5/25/0.5 ACN/H_2O/H_3PO_4 **or** Ethanol-0.5% H_3PO_4 using a blender during 3 minutes. After, the mixture was mixed during 30 minutes with magnetic stirrer. The mixture was filtered on filter paper (4-7µm). Then the mixture was diluted by 2 with water.

Cleanup with a 3mL/100mg AFFINIMIP® SPE Phenolics cartridge

Equilibration
- 3mL Acetonitrile
- 3mL Water

Loading
- Up to 80mL of loading solution

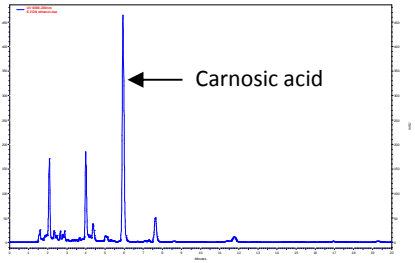
Washing of interferences
- 3mL Water / Acetonitrile (60/40 v/v)

Elution (E)
- 2mL Methanol -1% H_3PO_4

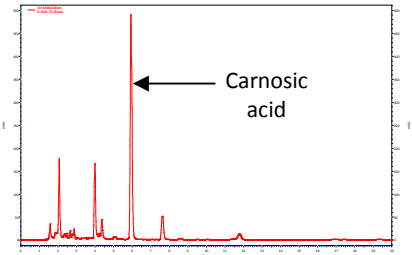
HPLC-UV Method

Column: Thermo Hypersil gold, 150mm x 4.6mm
Mobile phase: 65/35 (v/v) ACN/Water-0.5% H_3PO_4
Flow rate: 1mL/min
Detection: UV - 230nm
Injection volume: 5µL.

RESULTS



Chromatogram of a turkey containing 50ppm of Carnosic acid after clean-up with AFFINIMIP® SPE Phenolics. Extraction of the turkey with Ethanol-0.5% H3PO4



Chromatogram of a turkey containing 50ppm of Carnosic acid after clean-up with AFFINIMIP® SPE Phenolics. Extraction of the turkey with 74.5/25/0.5 ACN/H2O/H3PO4

Recovery yields obtained by both extraction solvent after AFFINIMIP® SPE Phenolics Clean-up.

Extraction solvent	Recoveries %
74.5/25/0.5 ACN/H_2O/H_3PO_4	>85%
Ethanol-0.5% H_3PO_4	>80%

Catalog number: FS103-02

ARTIFICIAL SWEETENERS IN WATER

PROTOCOL OF PURIFICATION

Purification with a 6mL/200mg AttractSPE™ HLB cartridge

Equilibration
- 5mL Methanol
- 5mL Water pH~2

Loading
50mL of water (adjusted to pH ~2)

Washing
10mL distilled water pH ~2

Drying 30min

Elution (E)
2mL Methanol

Analysis: LC-MS/MS

Acesulfame, Aspartame, Cyclamate, Neohespiridine dihydrochalcone, Saccharin, Sucralose

Catalog number: HLB-50.S.6.200

AttractSPE™ HLB

COCAINE AND MAIN METABOLITES IN WASTE WATER

PROTOCOL OF PURIFICATION

Waste water was adjusted to pH 2 with 37%HCl and filtered to form the loading solution.

Purification with a 6mL/500mg AttractSPE™ HLB cartridge

Equilibration
- 3mL Methanol
- 3mL Water pH~2

Loading
100mL Waste water pH ~6

Washing
- 3mL Methanol/Water (5/95)

Drying 15min

Elution (E)
2x4mL Methanol

Analysis: LC-MS/MS

Cocaine, benzoylecgonine and ecgonine methyl ester

Catalog number: HLB-50.S.6.500

MELAMINE IN MILK

PROTOCOL OF PURIFICATION

Sample preparation

Add 4mL water to 5g liquid infant formula or 1g dry infant formula. Shake during 10-20min with 20mL 50/50 ACN/Water and centrifuge for 10minutes at 3400 rpm.

The supernatant is the loading solution.

Purification with a 6mL/150mg AttractSPE™ SCX cartridge

Equilibration

- 5mL 0.1M NaOH in Acetonitrile
- 5mL 0.1M HCl in Acetonitrile
- 5mL Acetonitrile
- 5mL 4% Formic acid in Water

Loading

3mL 4% Formic acid in Water

2mL of loading solution

Washing of interferences

- 5mL Acetonitrile
- 5mL 0.2% diethyamine in Acetonitrile

Elution (E)

4mL 2% diethylamine in Acetonitrile

The elution fraction was filtered and then evaporated under nitrogen and dissolved in the mobile phase before HPLC analysis.

Analyse LC-MS/MS

Regulations for Melamine:
Codex alimentarius 35th CAC session (july 2012):
Maximum limit 0.15mg/kg for liquid infant milk

Catalog number: SCX-25.S.6.150

CYANURIC ACID IN MILK

PROTOCOL OF PURIFICATION

Sample preparation

Add 4mL water to 5g liquid infant formula or 1g dry infant formula. Shake during 10-20min with 20mL 50/50 ACN/Water and centrifuge for 10minutes at 3400 rpm.

The supernatant is the loading solution

Purification with a 6mL/150mg AttractSPE™ SAX cartridge

Equilibration

- 5mL 0.1M HCl in Acetonitrile
- 5mL 0.1M NaOH in Acetonitrile
- 5mL Acetonitrile
- 5mL 5% NH_4OH in Water

Loading

3mL 5% NH_4OH in Water

2mL of loading solution

Washing of interferences

- 5mL Acetonitrile

Elution (E)

2mL 4% Formic acid in Acetonitrile

The elution fraction was filtered and then evaporated under nitrogen and dissolved in the mobile phase before HPLC analysis.

Analyse LC-MS/MS

Catalog number: SAX-25.S.6.150

STEROIDS IN COW URINE

Analyzed steroids

Metabolite of Estrogens
Estra-1,3,5(10)-triene-3,17 -diol (E2)

Metabolites of Boldenone
5 -androst-1-en-17 -ol-3-one (M2)
5 - androst-1-en-17 -ol-3-one (M4)
1,4-androstadien-17 -ol-3-one (epiboldenone)

Metabolites of Nandrolone
Estr-4-en-17 -ol-3-one (17 - nandrolone)
5 -estran-3 ,17 -diol (E-aba)

Metabolites of Testosterone
5 -androstan-3 -ol-17-one (etiocholanolone)
5 -androstan-3 -ol-17-one (epiandrosterone)
Androst-4-en- 17 -ol-3-one (epiT)
5 -androstan-3 ,17 -diol (5-aba)

Other steroids:
5-androsten-3 -ol-17-one (DHEA)
5-androsten-3 ,17 - diol (androstenediol)

PROTOCOL OF PURIFICATION

Sample preparation

Five milliliters of urine were thawed at room temperature and submitted to an enzymatic deconjugation step using - glucuronidase from E. Coli at 37 ◦C overnight. Samples were then centrifuged at 1200×g (5 ◦C) for at least 10 min. 1mL of sodium acetate buffer 0.1M at pH 5.0 and 20µL of β-glucuronidase/sulfatase *Helix pomatia* enzyme solution at 1.0mg/mL in the same buffer were mixed thoroughly by vortex. The enzymatic reaction was carried out for 2h at 37°C to obtain the loading solution.

Purification with a 3mL/100mg AFFINIMIP® SPE Estrogens cartridge

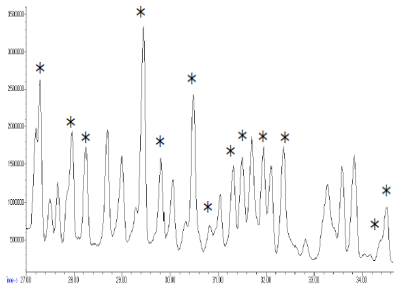
Equilibration
- 5mL Acetonitrile
- 5mL Water

Loading solution
loading solution

Washing of interferences
- 5mL Water/Acetonitrile (90/10)
- 5mL Water/Acetonitrile (80/20)

Elution (E)
3mL Methanol

RESULTS



Total Ion Chromatogram acquired in scan mode (GC-MS) : 2 µL injected of the eluted fraction from SPE-MIP after clean-up with AFFINIMIP® SPE Estrogens

Catalog number: FS104-02

A WIDE VARIETY OF DIOXINS - PCDD/Fs, PCBs, PBDEs, PBDD/Fs, OH-BDEs, OH-CB/BDE

Recovery and RSD of some native dioxins and hydroxylated dioxins analyzed in the publication

Analyte	Recovery %	RSD
2,3,7,8-TCDF	73	14.7
2,3,7,8-TCDD	81	17.5
1,2,3,7,8-PeCDD	86	15.6
1,2,3,4,7,8-HxCDF	88	15.4
1,2,3,6,7,8-HxCDD	78	9.6
1,2,3,4,6,7,8-HeCDF	74	13.3
CB28	79	12.7
CB 52	82	14.7
2,3,7,8-TBDD	76	12.7
1,2,3,4,6,7,8-HpBDD	77	15.8
5-MeOBDE99	82	13.9
4-MeOCB101	86	13.2
BDE12	82	16.1
BDE25	82	15.9
BDE35	80	13.4
BDE118	85	11.7
4-OH-CB19	64	17.8
4-OH-CB50	75	16.0
4-OH-CB106	72	14.5
4-OH-CB159	80	12.3
4-OH-CB172	74	12.3
3-OH-BDE28	74	11.6
3-OH-BDE47	80	17.8
6-OH-BDE137	82	14.7
6-OH-BDE-180	73	12.7

PROTOCOL OF CLEANUP

Cleanup with a 3mL/100mg AFFINIMIP® SPE Phenolics cartridge

Equilibration

- 6mL Methanol – 2% Acetic acid
- 6mL Methanol
- 6mL Dichloromethane

Loading

- Loading solution based on dichloromethane

Washing of interferences

- 20mL Dichloromethane (elution of neutral compounds)

Elution of phenolic compounds

- 20mL dichloromethane – 10% formic acid

Publications

Data extracted from the article Simultaneous separation of chlorinated/brominated dioxins, polychlorinated biphenyls, polybrominated diphenylethers and their methoxylated derivatives from hydroxylated analogues on molecularly imprinted polymers prior to gas/liquid chromatography and mass spectrometry, M. Roszko, K. Szymczyk, R. Jędrzejczak, *Talanta* 144, 171-183, 2015..

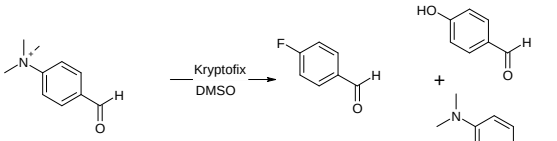
Catalog number: FS103-02

RADIOTRACERS PURIFICATION

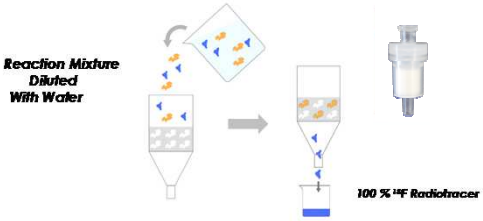
AFFINIMIP® SPE ^{18}F - Aromatic Nucleophilic Substitution

SYNTHESIS AND PURIFICATION OF 4-Fluorobenzaldehyde (FBA)

Aromatic nucleophilic substitution is widely used to synthesize fluorous radiotracers. Due to the short lifetime of ^{18}F radiotracers, the purification is a key step of the synthesis. It must be fast and effective to ensure a high radiochemical purity. This application note shows the effectiveness of the purification method for FBA using a AFFINIMIP® SPE ^{18F} Aromatic Nucleophilic Substitution cartridge.



Precursor Radiotracer By-products



PROTOCOL OF PURIFICATION

At 95°C were mixed 5 mg of ammonium salt, 6.7 mg of Kryptofix 2.2.2 and 1 mg of potassium fluoride in 400 µL of DMSO. After 15 minutes and the cooling of the reaction mixture, 2 mL of water were added to obtain the loading solution (L).

Cleanup with a AFFINIMIP® SPE ^{18F} Aromatic Nucleophilic Substitution cartridge

Equilibration
5mL Acetonitrile

Loading
Loading solution

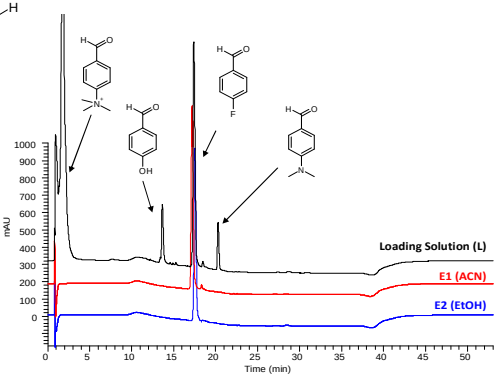
Washing of interferences
5mL of 80-20 Water-ACN

Drying 30s

Elution (E)
•1-2mL ACN

RESULTS

Recovery of more than 95 % of the fluorous radiotracer was obtained without any contamination of other identified compounds (phenolic and dimethylaminobenzylic compounds, precursor and fluoride). More than 95 % of Kryptofix 2.2.2 was also eliminated. (MS control)



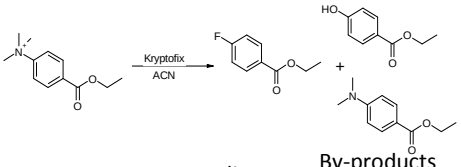
Fluoration of 4-Trimethylammoniumbenzaldehyde Tosylate in DMSO. Chromatograms obtained before (black) and after AFFINIMIP® SPE ^{18F} Aromatic Nucleophilic Substitution Clean-up (E1 : red) and after AttractSPE™ HLB Clean-up (E2 : blue)

Catalog number: RP100-01

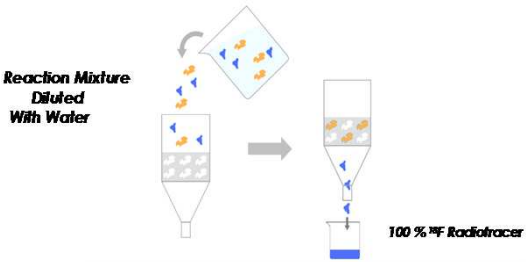
AFFINIMIP® SPE ^{18}F - Aromatic Nucleophilic Substitution

SYNTHESIS AND PURIFICATION OF Ethyl 4-Fluorobenzoate

Aromatic nucleophilic substitution is widely used to synthesize fluorous radiotracers. Due to the short lifetime of ^{18}F radiotracers, the purification is a key step of the synthesis. It must be fast and effective to ensure a high radiochemical purity. This application note shows the effectiveness of the purification method for Ethyl 4-Fluorobenzoate radiotracers using a AFFINIMIP® SPE ^{18F} Aromatic Nucleophilic Substitution cartridge.



Precursor Radiotracer By-products



PROTOCOL OF PURIFICATION

At 95°C were mixed 5 mg of ammonium salt, 6.7 mg of Kryptofix 2.2.2 and 1 mg of potassium fluoride in 400 µL of ACN. After 15 minutes and the cooling of the reaction mixture, 2 mL of water were added to obtain the loading solution.

Cleanup with a AFFINIMIP® SPE ^{18F} Aromatic Nucleophilic Substitution cartridge

Equilibration
5mL Acetonitrile

Loading
Loading solution

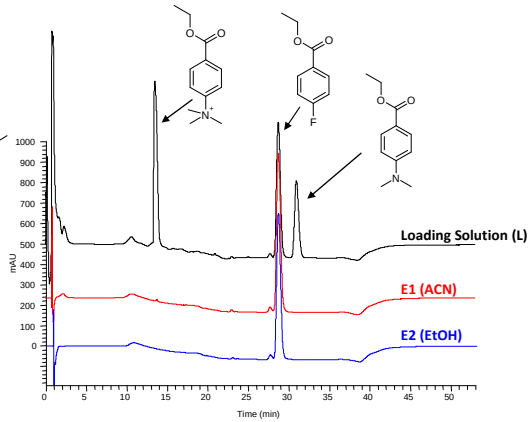
Washing of interferences
5mL of 80-20 Water-ACN

Drying 30s

Elution (E)
•1-2mL ACN

RESULTS

Recovery of more than 95 % of the fluorous radiotracer was obtained without any contamination of other identified compounds (phenolic and dimethylaminobenzylic compounds, precursor and fluoride). More than 95 % of Kryptofix 2.2.2 was also eliminated. (MS control)

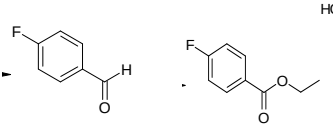


Fluoration of Ethyl 4-Trimethylammoniumbenzoate iodide in DMSO. Chromatograms obtained before (black) and after AFFINIMIP® SPE ^{18F} Aromatic Nucleophilic Substitution Clean-up (E1 : red) and after AttractSPE™ HLB Clean-up (E2 : blue)

Catalog number: RP100-01

Use of AttractSPE™ HLB cartridge to get the radiotracer in Ethanol

Having the radiotracer in Ethanol at the end of the radiosynthesis can be realized with an AttractSPE™ HLB cartridge.
This procedure must be fast and effective to ensure a high radiochemical purity.



Ethyl 4-Fluorobenzoate and 4-Fluorobenzaldehyde were respectively previously obtained in an acetonitrile solution noted E1.

PROTOCOL OF PURIFICATION

Cleanup with a AttractSPE™ HLB reversible cartridge

Equilibration

2mL Ethanol
2mL Water

Loading

Load with the acetonitrile elution E1 diluted with 15mL water

Drying 30 s

Elution

Elute the fluorous radiotracer with 1-2mL of Ethanol until dryness (E2)

HPLC-Fluorescence Method

Column: Hypersil Gold column 50mm x 2.1mm, 1.9 µm
Mobile phase:

Time (min)	% (0.1 % HCOOH Water)	% ACN
0	100	0
3	100	0
15	70	30
32	70	30
33	100	0
53	100	0

Flow rate: 0.2mL/min
Injection volume: 10µL. – UV 235nm

RESULTS

Recovery of more than 95 % of the fluorous radiotracer was obtained

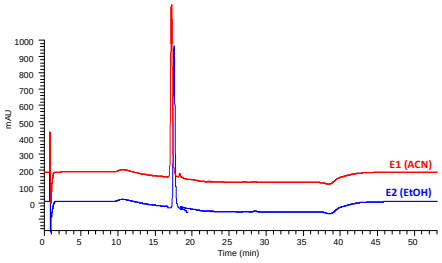


Figure 1. Obtention of 4-Fluorobenzaldehyde in Ethanol. Chromatograms obtained before (red) and after AttractSPE™ HLB Clean-up (E2 : blue)

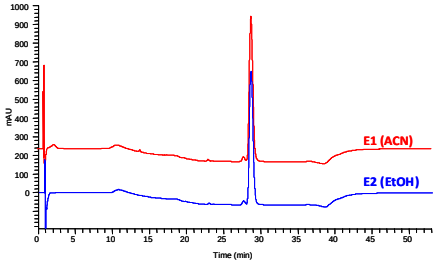


Figure 2. Obtention of Ethyl 4-Fluorobenzoate in Ethanol. Chromatograms obtained before (red) and after AttractSPE™ HLB Clean-up (E2 : blue)

Conclusion

The use of AttractSPE™ HLB allows to get the radiotracer in a minimum of Ethanol.

POCIS

Be selective

Food / Feed Safety

Environment

Cosmetics

Pharmaceutical R&D

MONITORING OF GLYPHOSATE - AMPA WITH A PASSIVE SAMPLER

Passive Sampling with POCIS

Passive sampling enables the monitoring of contaminants in water (surface water, groundwater, coastal water...) for a long period (days or weeks). An average of the concentration of this contaminant is measured.

For hydrophilic organic compounds, the Polar Organic Chemical Integrative Sampler (POCIS) is designed to provide the time weighted average (TWA) concentration of chemicals during the sampling period.

The POCIS consists of a solid sorbent contained between two microporous membranes. The sorbent collects the contaminant in water. Each sorbent may have a retention for specific contaminant or a family of contaminant.

AFFINIMIP® POCIS Glyphosate

AFFINIMIP® POCIS Glyphosate enables the sampling of Glyphosate and AMPA in water (Groundwater, geothermal, mineral...).

Then the powder is collected in an empty SPE column for the extraction of Glyphosate and AMPA

PROTOCOL OF EXTRACTION

Extraction of collected Glyphosate and AMPA from AFFINIMIP® POCIS Glyphosate with a SPE

Washing of interferences (optional)

Water

Extraction of the analytes (E)

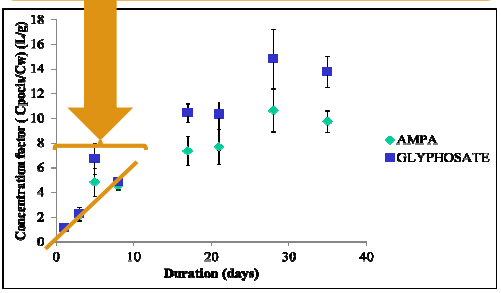
HCl solution (100mM)

The extraction solution is then evaporated and reconstituted with water prior analysis

RESULTS

Laboratory sampling rates estimation for AMPA and glyphosate using the AFFINIMIP® POCIS Glyphosate

Sampling rates: 130mL/day/200mg AFFINIMIP® POCIS Glyphosate in agreement with other pesticides in classical POCIS.



Mineral water (pH = 7) fortified at 500ng/L of AMPA and glyphosate. Concentrations kept constant during whole experiment.

Pesticides concentration in the tank, temperature, TOC and conductivity monitored during the experimental period to verify the stability of physico-chemical conditions in water.

Catalog number: POCIS-GLY.90.55.A.1

PRODUCT LIST

Be selective

Food / Feed Safety

Environment

Cosmetics

Pharmaceutical R&D

Products	Designation	Description	25c/box	50 c/box
Multi-mycotoxins	AFFINIMIP® SPE Multimyco LCMSMS	3mL for Multimycotoxin analyses	FS118-02	FS118-03
		6mL for Multimycotoxin analyses	FS118-02B	FS118-03B
Zearalenone & Fumonisins	AFFINIMIP® SPE FumoZON	3mL for Zearalenone and Fumonisins	FS109-02	FS109-03
Patulin	AFFINIMIP® SPE Patulin	3mL – 100mg for Patulin	FS102-02	FS102-03
		6mL – 200mg for Patulin	FS102-02B-200mg	FS102-03B-200mg
	AFFINIMIP® SPE Patulin & Pectinase kit	Kit of 3mL cartridges for Patulin + 50mL Pectinase enzyme solution	FS102-02K	FS102-03K
		Kit of 6mL - 200mg cartridges for Patulin in dried apple + 50mL Pectinase enzyme solution	FS102-02KB-200mg	FS102-03KB-200mg
Ochratoxin A	AFFINIMIP® SPE Ochratoxin A	3mL for Ochratoxin A	FS101-02	FS101-03
		6mL for Ochratoxin A	FS101-02B	FS101-03B
DON	AFFINIMIP® SPE Deoxynivalenol	6mL -100mg for Deoxynivalenol in food and babyfood	FS117-02B	FS117-03B
		6mL – 200mg for Deoxynivalenol in feed	FS117-02B-200mg	FS117-03B-200mg
Zearalenone	AFFINIMIP® SPE Zearalenone	3mL for ZON	FS100-02	FS100-03
Pectinase		50 mL Pectinase enzyme solution	REA-001-50mL	

Products	Designation	Description	25c/box	50 c/box
Bisphenol A and analogues	AFFINIMIP® SPE Bisphenols	3mL for Bisphenols (PP)	FS106-02	FS106-03
		6mL for Bisphenols (PP)	FS106-02B	FS106-03B
		6mL for Bisphenols (Glass)	FS106-02G	FS106-03G
Estrogens	AFFINIMIP® SPE Estrogens	1mL for Estrogens	FS104-02A	FS104-03A
		3mL for Estrogens	FS104-02	FS104-03
		96 well plate for estrogens– 1/pk	FS104-1.96W	
Catecholamines	AFFINIMIP® SPE Catecholamines	3mL for Catecholamines	DG100-02	DG100-03
		1mL for Catecholamines	DG100-02A	DG100-03A
Metanephrines	AFFINIMIP® SPE Metanephrines	3mL for Metanephrines	DG101-02	DG101-03
		1mL for Metanephrines	DG101-02A	DG101-03A
Picloram, Aminopyralid, Clopyralid	AFFINIMIP® SPE Picolinic Herbicides	3mL for Picolinic acid based herbicides	FS115-02	FS115-03
Glyphosate, AMPA	AFFINIMIP® SPE Glyphosate -AMPA	3mL for Glyphosate and AMPA	FS113-02	FS113-03
		6mL for Glyphosate and AMPA	FS113-02B	FS113-03B
NNAL	AFFINIMIP® SPE NNAL	3mL for NNAL	DG103-02	DG103-03
		96 well plate – 1/pk	DG103-1.96W	
Amphetamines	AFFINIMIP® SPE Amphetamines	3mL for Amphetamines derivatives	DG102-02	DG102-03
Chloram--phenicol	AFFINIMIP® SPE Chloramphenicol	1mL for Chloramphenicol	FS110-02A	FS110-03A
		3mL for Chloramphenicol	FS110-02	FS110-03
Tamoxifen	AFFINIMIP® SPE Tamoxifen	3mL for Tamoxifen	PH101-02	PH101-03
Tetracyclines	AFFINIMIP® SPE Tetracyclines	1mL for Tetracyclines	FS112-02A	FS112-03A
		3mL for Tetracyclines	FS112-02	FS112-03
Zeranol Residues	AFFINIMIP® SPE Zeranol Residues	3mL for Zeranol Residues	FS105-02	FS105-03
Phenolics	AFFINIMIP® SPE Phenolics	3mL for Phenolic compounds	FS103-02	FS103-03
PAHs	AFFINIMIP® SPE PAHs	3mL for PAHs	FS119-02	FS119-03

Product	Description	Reference	Number of cartridges
Reversible cartridges (2mL) 	SPE cartridges compatible with automates for purification of radiotracers issued from Aromatic Nucleophilic Substitution	RP100-01	10
	SPE cartridges compatible with automates for purification of radiotracers issued from Aromatic Nucleophilic Substitution	RP100-02	25
Reversible cartridges (0.7mL) 	SPE cartridges compatible with automates for purification of radiotracers issued from Aromatic Nucleophilic Substitution	RP100A-01	10
	SPE cartridges compatible with automates for purification of radiotracers issued from Aromatic Nucleophilic Substitution	RP100A-02	25

Format, amount	#/box	AttractSPE™ HLB	AttractSPE™ SCX	AttractSPE™ WCX	AttractSPE™ SAX	AttractSPE™ WAX	AttractSPE™ DVB
1mL, 30mg	100	HLB-100.S.1.30	SCX-100.S.1.30	WCX-100.S.1.30	SAX-100.S.1.30	WAX-100.S.1.30	DVB-100.S.1.30
3mL, 60mg	50	HLB-50.S.3.60	SCX-50.S.3.60	WCX-50.S.3.60	SAX-50.S.3.60	WAX-50.S.3.60	DVB-50.S.3.60
	100	HLB-100.S.3.60	SCX-100.S.3.60	WCX-100.S.3.60	SAX-100.S.3.60	WAX-100.S.3.60	DVB-100.S.3.60
6mL, 200mg	25	HLB-25.S.6.200	SCX-25.S.6.200	WCX-25.S.6.200	SAX-25.S.6.200	WAX-25.S.6.200	DVB-25.S.6.200
	50	HLB-50.S.6.200	SCX-50.S.6.200	WCX-50.S.6.200	SAX-50.S.6.200	WAX-50.S.6.200	DVB-50.S.6.200
	100	HLB-100.S.6.200	SCX-100.S.6.200	WCX-100.S.6.200	SAX-100.S.6.200	WAX-100.S.6.200	DVB-100.S.6.200
6mL, 500mg	25	HLB-25.S.6.500	SCX-25.S.6.500	WCX-25.S.6.500	SAX-25.S.6.500	WAX-25.S.6.500	DVB-25.S.6.500
	50	HLB-50.S.6.500	SCX-50.S.6.500	WCX-50.S.6.500	SAX-50.S.6.500	WAX-50.S.6.500	DVB-50.S.6.500
	100	HLB-100.S.6.500	SCX-100.S.6.500	WCX-100.S.6.500	SAX-100.S.6.500	WAX-100.S.6.500	DVB-100.S.6.500
12mL, 500mg	25	HLB-25.S.12.500	SCX-25.S.12.500	WCX-25.S.12.500	SAX-25.S.12.500	WAX-25.S.12.500	DVB-25.S.12.500
20mL, 1g	25	HLB-25.S.20.1g	SCX-25.S.20.1g	WCX-25.S.20.1g	SAX-25.S.20.1g	WAX-25.S.20.1g	DVB-25.S.20.1g
96 wells Plate, 30mg	1	HLB-1.96W.30	SCX-1.96W.30	WCX-1.96W.30	SAX-1.96W.30	WAX-1.96W.30	DVB-1.96W.30
Reversible 0.7mL, 30mg	25	HLB-25.REV.1.N10	SCX-25.REV.1.N10	WCX-25.REV.1.N10	SAX-25.REV.1.N10	WAX-25.REV.1.N10	DVB-25.REV.1.N10
	50	HLB-50.REV.1.N10	SCX-50.REV.1.N10	WCX-50.REV.1.N10	SAX-50.REV.1.N10	WAX-50.REV.1.N10	DVB-50.REV.1.N10
Reversible 0.7mL, 100mg (200mg for DVB)	25	HLB-25.REV.1.F	SCX-25.REV.1.F	WCX-25.REV.1.F	SAX-25.REV.1.F	WAX-25.REV.1.F	DVB-25.REV.1.F
	50	HLB-50.REV.1.F	SCX-50.REV.1.F	WCX-50.REV.1.F	SAX-50.REV.1.F	WAX-50.REV.1.F	DVB-50.REV.1.F
Reversible 2mL, 225mg	25	HLB-25.REV.2.N10	SCX-25.REV.2.N10	WCX-25.REV.2.N10	SAX-25.REV.2.N10	WAX-25.REV.2.N10	DVB-25.REV.2.N10
	50	HLB-50.REV.2.N10	SCX-50.REV.2.N10	WCX-50.REV.2.N10	SAX-50.REV.2.N10	WAX-50.REV.2.N10	DVB-50.REV.2.N10

Cartridges format, Sorbent amount	#/box	AttractSPE™ SAX-HCO3	AttractSPE™ PS-H	AttractSPE™ PS-Ag	AttractSPE™ PS-Ba	AttractSPE™ IDA
1mL	100	SAX-HCO3-100.S.1.30		PSAg-100.S.1.30	PSBa-100.S.1.30	IDA-100.S.1.30
3mL, 60mg	25	SAX-HCO3-25.S.3.60	PSH-25.S.3.60	PSAg-25.S.3.60	PSBa-25.S.3.60	IDA-25.S.3.60
	50	SAX-HCO3-50.S.3.60	PSH-50.S.3.60	PSAg-50.S.3.60	PSBa-50.S.3.60	IDA-50.S.3.60
6mL, 200mg	25	SAX-HCO3-25.S.6.200	PSH-25.S.6.200	PSAg-25.S.6.200	PSBa-25.S.6.200	IDA-25.S.6.200
	50	SAX-HCO3-50.S.6.200	PSH-50.S.6.200	PSAg-50.S.6.200	PSBa-50.S.6.200	IDA-50.S.6.200
6mL, 500mg	25	SAX-HCO3-25.S.6.500	PSH-25.S.6.500	PSAg-25.S.6.500	PSBa-25.S.6.500	IDA-25.S.6.500
	50	SAX-HCO3-50.S.6.500	PSH-50.S.6.500	PSAg-50.S.6.500	PSBa-50.S.6.500	IDA-50.S.6.500
96 wells Plate	1	SAX-HCO3-1.96W.30	PSH-1.96W.30			IDA-1.96W.30
Reversible 0.7mL, 30mg	25	SAX-HCO3-25.REV.1.F	PSH-25.S.REV.1.F			IDA-25.REV.1.N10
	50	SAX-HCO3-50.REV.1.F	PSH-50.S.REV.1.F			IDA-50.REV.1.N10
Reversible 0.7mL, 100mg	25	SAX-HCO3-25.REV.2.F	PSH-25.S.REV.2.F	PSAg-25.S.REV.1.F For 400mg	PSBa-25.S.REV.1.F For 400mg	IDA-25.REV.1.F
	50	SAX-HCO3-50.REV.2.F	PSH-50.S.REV.2.F	PSAg-50.S.REV.1.F for 400mg	PSBa-50.S.REV.1.F For 400mg	IDA-50.REV.1.F
Reversible 2mL, 800mg	25	SAX-HCO3-100.S.1.30				IDA-25.REV.2.F
	50	SAX-HCO3-25.S.3.60	PSH-25.S.3.60			IDA-50.REV.2.F

AttractSPE™ Carbon based SPE - Product list

Product	Vol	Sorbent	25 cartridges/box	50 cartridges/box
AttractSPE™ Carbon	6mL	500mg	Carb-25.S.6.500	Carb-50.S.6.500
AttractSPE™ Carbon/PSA	3mL	250mg/ 250mg	CarbPSA-25.S.3.250.250	CarbPSA-50.S.3.250.250
	6mL	500mg/ 500mg	CarbPSA-25.S.6.500.500	CarbPSA-50.S.6.500.500
AttractSPE™ Carbon/Amine	6mL	500mg/ 500mg	CarbNH2-25.S.6.500.500	CarbNH2-50.S.6.500.500

AttractSPE™ LipRem

Cartridges format, Sorbent amount	#/box	AttractSPE™ LipRem
1mL, 20mg	100	LipRem-100.S.1.20
3mL, 60mg	25	LipRem-25.S.3.50
	50	LipRem-50.S.3.50
6mL, 100mg	25	LipRem-25.S.6.100
	50	LipRem-50.S.6.100
96 wells Plate	1	LipRem-1.96W.20
Reversible 0.7mL, 100mg	25	LipRem-1.REV.1.F
	50	LipRem-1.REV.1.F

		Non polar sorbents			Polar sorbents			
Cartridges format, Sorbent amount	#/box	SilactSPE™ C18	SilactSPE™ C8	SilactSPE™ Phenyl	SilactSPE™ Silica	SilactSPE™ Cyano	SilactSPE™ Florisil	SilactSPE™ Florisil PR
1mL, 50mg	100	C18-100.S.1.50	C8-100.S.1.50	Phe-100.S.1.50	Si-100.S.1.50	CN-100.S.1.50	Flo-100.S.1.50	FloPR-100.S.1.50
1mL, 100mg	100	C18-100.S.1.100	C8-100.S.1.100	Phe-100.S.1.100	Si-100.S.1.100	CN-100.S.1.100	Flo-100.S.1.100	FloPR-100.S.1.100
3mL, 200mg	50	C18-50.S.3.200	C8-50.S.3.200	Phe-50.S.3.200	Si-50.S.3.200	CN-50.S.3.200	Flo-50.S.3.200	FloPR-50.S.3.200
3mL, 500mg	50	C18-50.S.3.500	C8-50.S.3.500	Phe-50.S.3.500	Si-50.S.3.500	CN-50.S.3.500	Flo-50.S.3.500	FloPR-50.S.3.500
6mL, 500mg	50	C18-50.S.6.500	C8-50.S.6.500	Phe-50.S.6.500	Si-50.S.6.500	CN-50.S.6.500	Flo-50.S.6.500	FloPR-50.S.6.500
6mL, 1g	50	C18-50.S.6.1g	C8-50.S.6.1g	Phe-50.S.6.1g	Si-50.S.6.1g	CN-50.S.6.1g	Flo-50.S.6.1g	FloPR-50.S.6.1g
6mL, 2g	50	C18-50.S.6.2g	C8-50.S.6.2g	Phe-50.S.6.2g	Si-50.S.6.2g	CN-50.S.6.2g	Flo-50.S.6.2g	FloPR-50.S.6.2g
12mL, 2g	20	C18-20.S.12.2g	C8-20.S.12.2g	Phe-20.S.12.2g	Si-20.S.12.2g	CN-20.S.12.2g	Flo-20.S.12.2g	FloPR-20.S.12.2g
Reversible 0.7mL, 200mg	25	C18-25.REV.1.200	C8-25.REV.1.200	Phe-25.REV.1.200	Si-25.REV.1.200	CN-25.REV.1.200	Flo-25.REV.1.200	FloPR-25.REV.1.200
Reversible 2mL, 750mg	25	C18-25.REV.2.750	C8-25.REV.2.750	Phe-25.REV.2.750	Si-25.REV.2.750	CN-25.REV.2.750	Flo-25.REV.2.750	FloPR-25.REV.2.750

For other formats, please contact us

		Polar sorbents			Others sorbents			
Cartridges format, Sorbent amount	#/box	SilactSPE™ Alumina Acidic	SilactSPE™ Alumina Neutral	SilactSPE™ Alumina Basic	SilactSPE™ Amine	SilactSPE™ PSA	SilactSPE™ Carbonate	SilactSPE™ Hydroxy Apatatite
1mL, 50mg	100	AluA-100.S.1.50	AluN-100.S.1.50	AluB-100.S.1.50	NH2-100.S.1.50	PSA-100.S.1.50	CO3-100.S.1.50	HAp-100.S.1.50
1mL, 100mg	100	AluA-100.S.1.100	AluN-100.S.1.100	AluB-100.S.1.100	NH2-100.S.1.100	PSA-100.S.1.100	CO3-100.S.1.100	
3mL, 200mg	50	AluA-50.S.3.200	AluN-50.S.3.200	AluB-50.S.3.200	NH2-50.S.3.200	PSA-50.S.3.200	CO3-50.S.3.200	HAp-50.S.3.200
3mL, 500mg	50	AluA-50.S.3.500	AluN-50.S.3.500	AluB-50.S.3.500	NH2-50.S.3.500	PSA-50.S.3.500	CO3-50.S.3.500	
6mL, 500mg	50	AluA-50.S.6.500	AluN-50.S.6.500	AluB-50.S.6.500	NH2-50.S.6.500	PSA-50.S.6.500	CO3-50.S.6.500	HAp-50.S.6.500
6mL, 1g	50	AluA-50.S.6.1g	AluN-50.S.6.1g	AluB-50.S.6.1g	NH2-50.S.6.1g	PSA-50.S.6.1g	CO3-50.S.6.1g	
6mL, 2g	50	AluA-50.S.6.2g	AluN-50.S.6.2g	AluB-50.S.6.2g	NH2-50.S.6.2g	PSA-50.S.6.2g	CO3-50.S.6.2g	
12mL, 2g	20	AluA-20.S.12.2g	AluN-20.S.12.2g	AluB-20.S.12.2g	NH2-20.S.12.2g	PSA-20.S.12.2g	CO3-20.S.12.2g	
Reversible 0.7mL, 200mg	25	AluA-25.REV.1.200	AluN-25.REV.1.200	AluB-25.REV.1.200	NH2-25.REV.1.200	PSA-25.REV.1.200	CO3-25.REV.1.200	HAp-50.REV.1.F
Reversible 2mL, 750mg	25	AluA-25.REV.2.750	AluN-25.REV.2.750	AluB-25.REV.2.750	NH2-25.REV.2.750	PSA-25.REV.2.750	CO3-25.REV.2.750	

For other formats, please contact us

SPE for Polycyclic Aromatic Hydrocarbons (PAHs) in soil

Product	Vol	Sorbent	25 cartridges/box	50 cartridges/box
SilactSPE™ CN/SiOH	3mL	500mg/1g	CNSiOH-25.S.3.500.1g	CNSiOH-50.S.3.500.1g
	6mL	500mg/1g	CNSiOH-25.S.6.500.1g	CNSiOH-50.S.6.500.1g
	6mL glass	500mg/1g	CNSiOH-25.G.6.500.1g	CNSiOH-50.G.6.500.1g

SilactSPE™ SLE

Cartridge volume	Sorbent	25 cartridges/box	50 cartridges/box
1mL	250mg	SLE-25.S.1.250	SLE-50.S.1.250
3mL	500mg	SLE-25.S.3.500	SLE-50.S.3.500
6mL	1g	SLE-25.S.6.1g	SLE-50.S.6.1g
15mL	3g	SLE-25.S.15.3g	SLE-50.S.15.3g
30mL	4.5g	SLE-25.S.30.4g	SLE-50.S.30.4g
70mL	14.5g	SLE-25.S.70.14g	SLE-50.S.70.14g

Fritted cartridges

Cartridge volume	SilactSPE™ Double fritted 100 cartridges	SilactSPE™ Single fritted 100 cartridges
1mL	0-100.S.1.2F	0-100.S.1.1F
3mL	0-100.S.3.2F	0-100.S.3.1F
6mL	0-100.S.6.2F	0-100.S.6.1F
15mL	0-100.S.15.2F	0-100.S.15.1F
25mL	0-100.S.25.2F	0-100.S.25.1F
60mL	0-100.S.60.2F	0-100.S.60.1F

Qcleanup™ EXTRACTION SALTS

QuEChERS methods	Description	Pouches / box	Product reference
Original method	4g $MgSO_4$ 1g NaCl	50	EXT.ORL.50
EN 15662	1g Trisodium citrate Dihydrate 0.5g Disodium hydrogencitrate sesquihydrate 1g NaCl and 4g $MgSO_4$	50	EXT.EN.50
AOAC 2007.01	1.5g Sodium Acetate and 6g $MgSO_4$	50	EXT.AOAC.50

Qcleanup™ DISPERSIVE SPE PRODUCTS

Method	Description	Nber/box	Product reference
	For General Fruits & Vegetables		
EN 15662	150mg $MgSO_4$ + 25mg PSA	100 tubes of 2mL	dSPE.EN.GFV.100.2
	900mg $MgSO_4$ + 150mg PSA	50 tubes of 15mL	dSPE.EN.GFV.50.15
AOAC 2007.01	150mg $MgSO_4$ + 50mg PSA	100 tubes of 2mL	dSPE.AOAC.GFV.100.2
	1200mg $MgSO_4$ + 400mg PSA	50 tubes of 15mL	dSPE.AOAC.GFV.50.15
	For Pigmented Fruits & Vegetables		
EN 15662	150mg $MgSO_4$ + 25mg PSA + 2.5mg CB	100 tubes of 2mL	dSPE.EN.PFV.100.2
	900mg $MgSO_4$ + 150mg PSA + 15mg CB	50 tubes of 15mL	dSPE.EN.PFV.50.15
AOAC 2007.01	150mg $MgSO_4$ + 50mg PSA + 50mg CB	100 tubes of 2mL	dSPE.AOAC.PFV.100.2
	1200mg $MgSO_4$ + 400mg PSA + 400mg CB	50 tubes of 15mL	dSPE.AOAC.PFV.50.15
	For Highly Pigmented and Fatty Fruits & Vegetables		
EN 15662	150mg $MgSO_4$ + 25mg PSA + 7.5mg CB	100 tubes of 2mL	dSPE.EN.HPFV.100.2
	900mg $MgSO_4$ + 150mg PSA + 45mg CB	50 tubes of 15mL	dSPE.EN.HPFV.50.15
AOAC 2007.01	150mg $MgSO_4$ + 50mg PSA + 50mg CB +50mg C18	100 tubes of 2mL	dSPE.AOAC.HPFV.100.2
	1200mg $MgSO_4$ + 400mg PSA + 400mg CB + 400mg C18	50 tubes of 15mL	dSPE.AOAC.HPFV.50.15
	For Fatty and waxed Fruits & Vegetables		
EN 15662	150mg $MgSO_4$ + 25mg PSA + 25mg C18	100 tubes of 2mL	dSPE.EN.FWFV.100.2
	900mg $MgSO_4$ + 150mg PSA + 150mg C18	50 tubes of 15mL	dSPE.EN.FWFV.50.15
AOAC 2007.01	150mg $MgSO_4$ + 50mg PSA + 50mg C18	100 tubes of 2mL	dSPE.AOAC.FWFV.100.2
	1200mg $MgSO_4$ + 400mg PSA + 400mg C18	50 tubes of 15mL	dSPE.AOAC.FWFV.50.15

Designation	Definition	Composition	Reference
AFFINIMIP® POCIS GLYPHOSATE	POCIS containing AFFINIMIP® GLYPHOSATE - AMPA for the retention of glyphosate and AMPA	1 POCIS	POCIS.GLY.90.55.A.1
		Kit of 10 POCIS + empty fritted cartridges	POCIS.GLY.90.55.kit.10
		Kit of 50 POCIS + empty fritted cartridges	POCIS.GLY.90.55.kit.50
AFFINIMIP® POCIS EDC	POCIS containing AFFINIMIP® Estrogens and AFFINIMIP® Bisphenols for the retention of endocrine disrupters such as natural/synthetic estrogens, Bisphenols...	1 POCIS	POCIS.EDC.90.55.A.1
		Kit of 10 POCIS + empty fritted cartridges	POCIS.EDC.90.55.kit.10
		Kit of 50 POCIS + empty fritted cartridges	POCIS.EDC.90.55.kit.50
Attract POCIS Pesticides	POCIS containing mixture of sorbent for the retention of several pesticides	1 POCIS	POCIS.PEST.90.55.A.1
		Kit of 10 POCIS + empty fritted cartridges	POCIS.PEST.90.55.kit.10
		Kit of 50 POCIS + empty fritted cartridges	POCIS.PEST.90.55.kit.50
Attract POCIS HLB	POCIS containing Attract HLB for the retention of pharmaceutical drug residues	1 POCIS	POCIS.HLB.90.55.A.1
		Kit of 10 POCIS + empty fritted cartridges	POCIS.HLB.90.55.kit.10
		Kit of 50 POCIS + empty fritted cartridges	POCIS.HLB.90.55.kit.50
CANISTER – 3 POCIS	Canister for 3 POCIS . Requires a holder	1 canister	CAN-3P.A.1
HOLDER – 3 POCIS	Holder for 3 POCIS	1 holder	HOLD-3P.A.1

POCIS

CANISTER – 3 POCIS

HOLDER – 3 POCIS

On-line SPE columns – Product list

Product	Product reference	Nber column	I.D. (mm)	Lenght (mm)
On-line AttractSPE™ HLB columns	OnlineSPE-HLB-1.2.20	1	2.1	20
	OnlineSPE-HLB-1.5.20	1	4.6	20
On-line AFFINIMIP® PHENOLICS columns	OnlineSPE-PHE-1.2.20	1	2.1	20
	OnlineSPE-PHE-1.5.20	1	4.6	20
On-line AFFINIMIP® ESTROGENS columns	OnlineSPE-EST-1.2.20	1	2.1	20
	OnlineSPE-EST-1.5.20	1	4.6	20

SPE ACCESSORIES – Product list

SPE Accessories	Designation	Definition	Reference
Manifold	SPE Vaccum Manifold	12-port model	ACC-MAN1
SPE Adapter & Reservoir kit	SPE Adapter & Reservoir kit	Kit of 12 reservoirs 60ml and adapters for use with 1,3 & 6 mL cartridges	ACC-AR1
Mini-Vap	Mini Evaporator/Concentrator	6 port Mini-Vap Evaporator/Concentrator for use with 1 to 250mL containers	ACC-VAP1
Mini PUMP	Mini vacuum pump	Laboport diaphragm vacuum mini pump, 5.5L/min	ACC-PUMP
Vacuum pump trap	SPE Vacuum pump trap kit	1L trap kit	ACC-TRAP

NONE EXHAUSTIVE LIST OF PUBLICATIONS AND POSTERS

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A new report of the French Health Agency (ANSES) on **assessment of the health risks associated with bisphenol A** (BPA) was published on 9 April 2013. Quantitative analysis of Bisphenol A in all liquid or solid food matrices were carried out by using AFFINIMIP® SPE Bisphenols (Analyses carried out by LABERCA and described in Annex 12 of Annexes of the report p132 (in french) and summary of the study).

➢Quantitative analysis of Bisphenol A in all liquid or solid food matrices were carried out by using AFFINIMIP® SPE Bisphenols (p132, Annex 12 of *Annexes to the report on the assessment of the risks associated with bisphenol A (BPA) for human health, and on toxicological data and data on the use of bisphenols S, F, M, B, AP, AF, and BADGE (In French)),* ANSES April 2013.

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About AFFINISEP

AFFINISEP is **a worldwide expert in purification and sample preparation applications as well as for the design and the development of intelligent polymers with** Molecularly Imprinted Polymers (MIP).
AFFINISEP is dedicated to the development of analytical applications in various fields such as water, biological fluids, food and feed analysis with a complete set of products and services for sample preparation and for passive sampling :

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