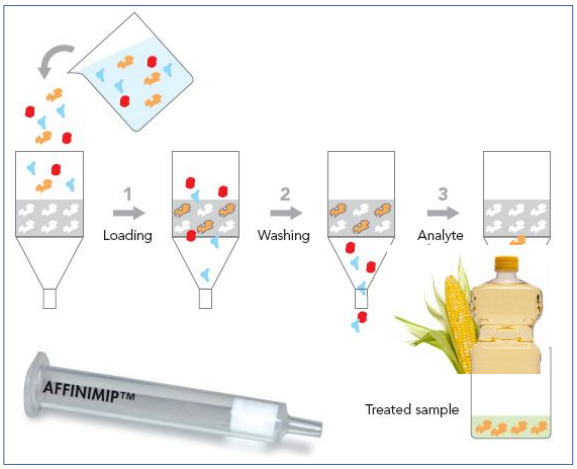
Fast and Selective Solid Phase Extraction of Zearalenone from Edible Corn Oil using AFFINIMIP® SPE Zearalenone cartridges



Background: "Corn germ oil and wheat germ oil make an important contribution to the zearalenone exposure"

Zearalenone [6-(10-hydroxy-6-oxo-trans-1-undecenyl)-β-resorcylic acid lactone; ZON] is a mycotoxin produced as a secondary metabolite of various Fusarium fungi (see figure 1). Zearalenone is known to cause estrogenic effects at relatively low levels, including infertility, reduced serum testosterone levels and sperm counts, reduced incidence of pregnancy, and change in progesterone levels. In addition, Zearalenone can delay the breeding process and cost the producer significant economic and physical losses.

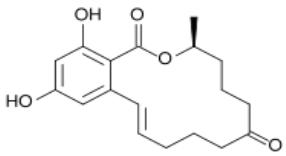


Figure 1. Chemical structure of Zearalenone, CAS N° 17924-92-4.

In Europe, Regulation (EC) 1126/2007 sets maximum levels for Zearalenone in foodstuffs, including corn oil (400µg/Kg). In addition, the European Food Safety Authority (EFSA) reported that "notably high levels of Zearalenone have been found in corn germ oil" and that "vegetable oil, especially corn germ oil and wheat germ oil, make an important contribution to the zearalenone exposure" (EFSA Journal 2011:9(6):2197).

How to clear the risk for consumers?

AFFINISEP has developed an efficient cleanup and preconcentration method based on molecularly imprinted polymers for the quantification of Zearalenone from corn oil using **AFFINIMIP®SPE Zearalenone**.

This method uses a very quick sample preparation by simply diluting corn oil in a solvent before cleanup.

Results

High recoveries and good repeatability

C° (µg/L)	Mean C° (µg/L)	Recoveries %	% RSD
400	397.4	99.3	8.0 (n=3)

Table 1. Recoveries of Zearalenone spiked at 400 µg/L in Corn Oil after cleanup by **AFFINIMIP®SPE Zearalenone**

Clean extracts at a broad range of contamination levels

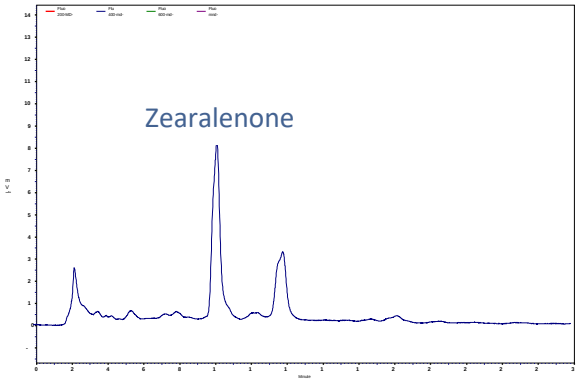


Figure 2. Chromatogram of Corn Oil spiked with Zearalenone at 200 µg/L obtained after cleanup by **AFFINIMIP®SPE Zearalenone**.

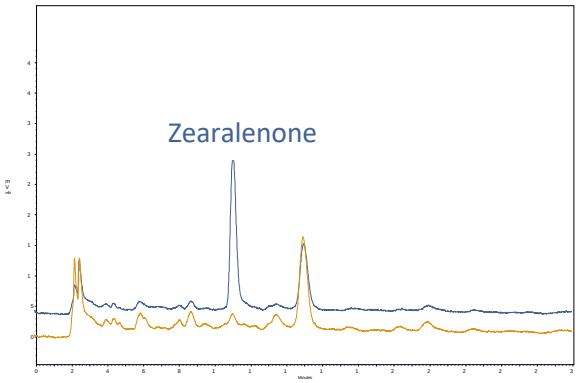


Figure3. Chromatograms of Corn Oil spiked with Zearalenone at 400µg/L (blue) or not spiked (orange) obtained after cleanup by **AFFINIMIP®SPE Zearalenone**.

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High recoveries at a broad range of contamination levels

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

Table 2. Recoveries of Zearalenone in Corn Oil at various contamination levels after **AFFINIMIP®SPE Zearalenone** cleanup.

Constant clean extracts for a clear quantification

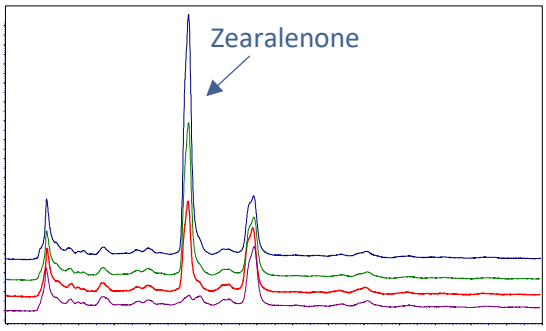

Figure4. Chromatograms obtained after cleanup by **AFFINIMIP®SPE Zearalenone** of Corn Oil spiked with Zearalenone at 200µg/L (red), 400µg/L (green), 600 µg/L (blue) or not spiked (purple).

Conclusion

Cleanup with **AFFINIMIP®SPE Zearalenone** provides clear and unambiguous results at a broad range of concentration levels. The tests performed demonstrate high recovery yields with a low background. In addition the sample preparation is very fast and simple.

Experimental conditions

Materials

All reagents and chemicals were ACS grade quality or better. Zearalenone used to spike the samples comes from POLYINTELL's mycotoxins standards portfolio. Edible corn oil was purchased in a local supermarket.

Preparation of samples prior to SPE

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

Solid phase extraction (SPE) protocol

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

- Condition the SPE Cartridge with 3mL of Diethyl Ether
- Load 3mL of the loading solution (eq. 1 mL of Maïze Oil sample)
- Wash the cartridge with 6 mL of Diethyl Ether
- Dry the cartridge during 30 seconds with vaccum
- Wash the cartridge with 6mL of 58/2/40 deionized water/acetic acid/ACN (v/v/v)
- Elute Zearalenone with 4mL of methanol (MeOH) containing 2% of acetic acid (v/v)

The elution fraction was then evaporated and dissolved in mobile phase. Alternatively, the elution may be diluted to a known volume by addition of water for further analysis. The SPE procedure lasted approximately 30 minutes.

Analysis

HPLC was performed on a ThermoFinnigan Spectra System with a Thermo Hypersil Gold C18 column (150mm x 4.6mm). Separation was carried out using a mobile phase of deionized water/MeOH (40/60, v/v) at a flow rate of 1mL/min. The detection system was a Jasco Model FP-2020 Fluorescence detector set to excitation/emission wavelengths of 275 and 450nm, respectively. The injection volume was 100µL.

References

Commission Regulation (EC) No. 1126/2007 of 28 September 2007, Official Journal of the European Union.

Commission Regulation (EC) No. 401/2006 of 23 February 2006, Official Journal of the European Union.

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