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Rapid simultaneous assay of 23 mycotoxines in a variety of food samples by UHPLC-MS/MS using fast polarity switching

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1. Introduction

Mycotoxins are contaminants in grains. For consumer food safety, quality control of food and beverages has to assay such contaminants. Rapid determination of the presence and then quantification of hazardous mycotoxins is essential. UHPLC-MS/MS offers the best combination of selectivity, sensitivity, and speed for detection of these compounds in complex matrices. In this study, a high throughput method for the quantification of 25 mycotoxins in various matrices was established.

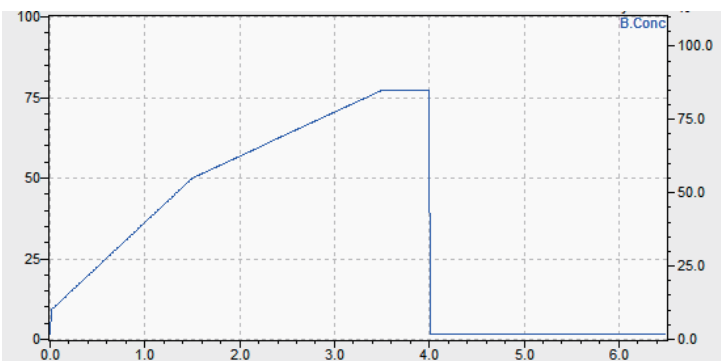
2. Methods and Materials

Sample preparation (modified QuEChERs)

Samples (5 g for grains or animal food + 10 mL of water or 10 g for fruits), were mixed with 10 mL of acetonitrile. After maceration, salts were added to allow phase separation.
Then the supernatant was 5-fold diluted with mobile A and the internal standard mix was added.

UHPLC conditions

System	: UFLCXR	
Column	: Phenomenex Kinetex XB-C18 50*2 mm 2.6µm	
Column temperature	: 50°C	
Mobile phase A	: Water + 0.5% acetic acid	
B	: Isopropanol + 0.5% acetic acid	
Flow rate	: 0.4 mL/min	
Time program	: Time (min)	Pump B Conc
	Initial	2
	0.01	10
	1.50	55
	3.50	85
	4.00	85
	4.01	2
	6.50	Stop
Injection vol.	: 20 µL	



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MS conditions (LCMS-8040)
Ionization : ESI, Positive and Negative MRM mode
Ion source temperatures: Desolvation line: 250°C
Heater Block : 500°C
Gases: Nebulization: 3 L/min
Drying : 15 L/min
MRM Transitions: Two transitions were selected for each mycotoxin
Dwell time: 9 msec
Pause time : 1 msec
Loop time : 0.27 sec (maximum)

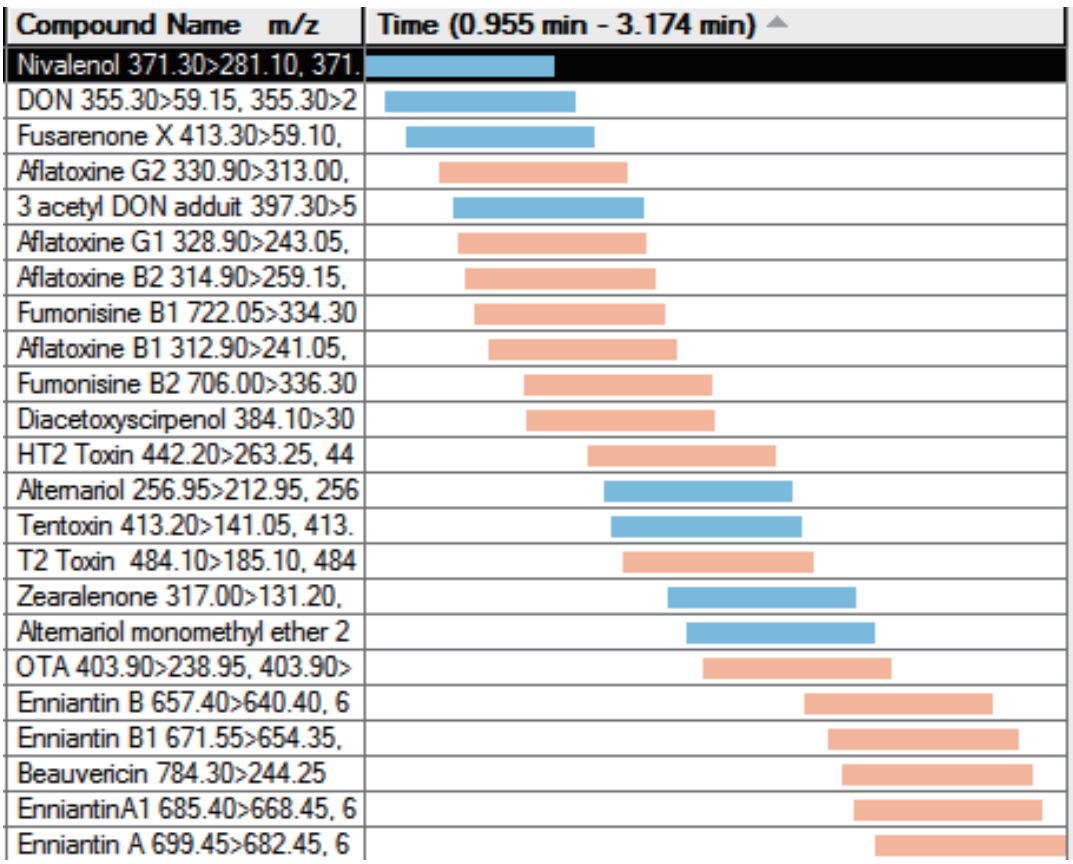

Fig. 1 MRM scheduling for positive (red) and negative (blue) ionization

3. Results

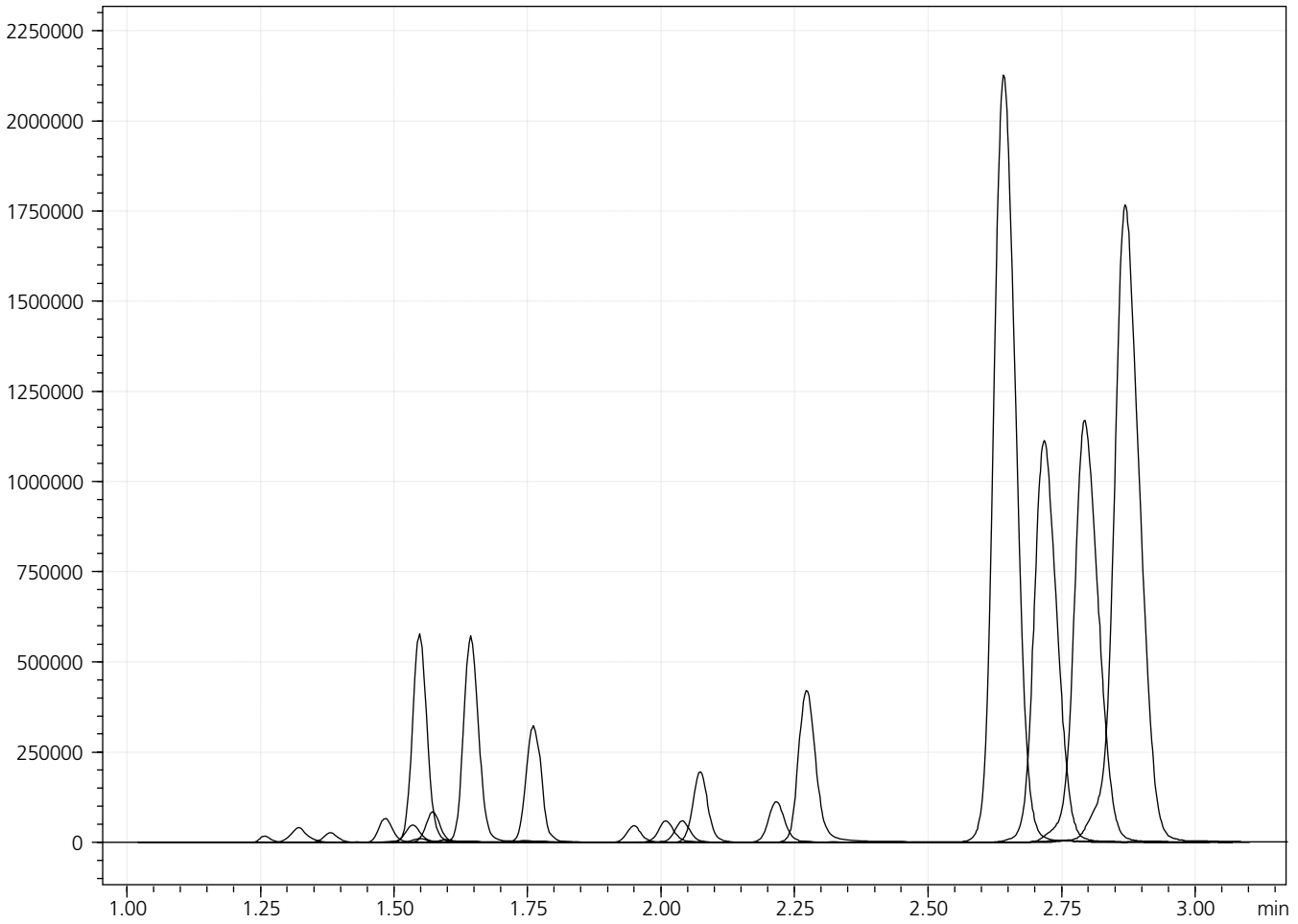

Fig. 2 typical chromatogram at 50 ppb (see Fig . 1 for elution order)

Specificity and matrix effect

The assay specificity was studied in various matrices to show the correlation of results in neat standards with matrix standard.
The concentration obtained was compared to the theoretical one. The specificity was validated for example in the following matrices : rice, maize, dry pastas, banana, muesli, wheat, carrot, apple compote, flour, etc....
The figure 3 shows the correlation data for exemplary mycotoxins in the cited matrices at different levels.
These data shows that the method gives accurate concentration results of the mycotoxins whatever the matrix assayed against neat solution standards. This suggests that the method is specific and free of significant matrix effect.

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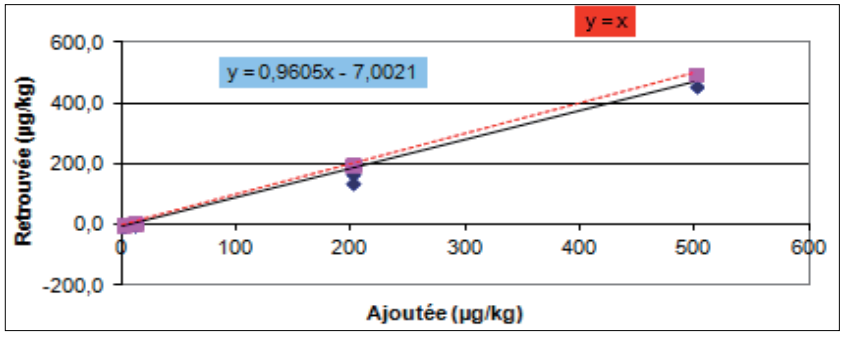
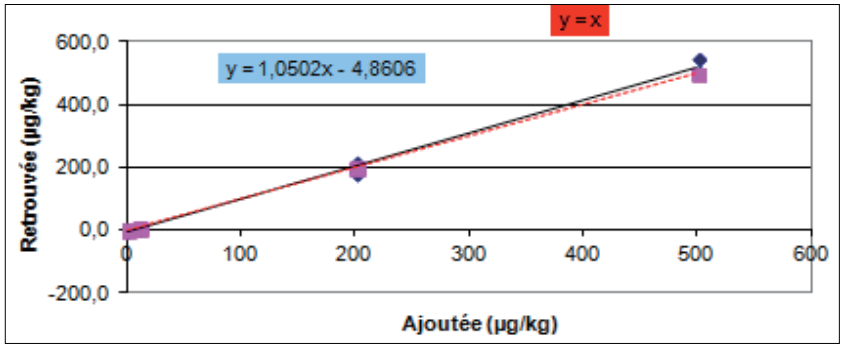
Aflatoxin B2



Ochratoxin A



Toxin T2

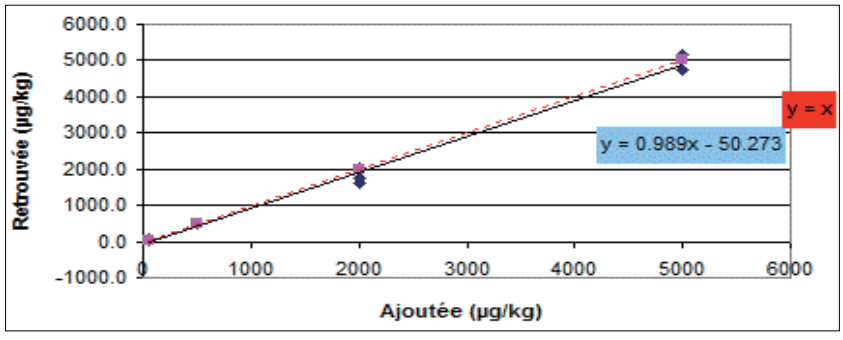


Fig. 3 Correlation data

4. Conclusions

- The fast polarity switching and the low electronic pause time allow simultaneous analysis of co-eluted compounds,
- The method is fast and accurate,
- The sample prepration and the selectivity of the method lowered the matrix affect, thus neat standards can be used for a variety of different samples. This increases system productivity.

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