Matrix of the Month

June 2015:

Deoxynivalenol in Oat Flakes via the Clean-Up Column DONeX

- manual and automated -

Do you have a special matrix that we should test for mycotoxins
Please let us know and write an e-mail to info@LCTech.de!

About Deoxynivalenol and DONeX

Deoxynivalenol, also known as vomitoxin, is a metabolic product of various fungi of the genus Fusarium (F. culmorum, F. graminearum) that occurs predominantly on cereals (wheat, barley, oats).

Mostly, this toxin is analysed via HPLC / UV detector or alternatively via HPLC / FLD in combination with post-column derivatisation or with LC / MS. In all three processes, thorough sample preparation increases the life of the analytical system and also the life of the HPLC column. In addition, pre-cleaning helps reduce interferences through matrix components and nearly halves the chromatography time of the HPLC analysis.

The DONEX clean-up column developed by LCTech excludes matrix interferences and herewith allows shorter chromatographies and reduces interfering matrix peaks. This results in better and faster chromatograms as well as in a higher measuring sensitivity.

Automated Sample Preparation with FREESTYLE SPE

The clean-up column is available in a 3 mL format and is thus suitable for automated processing in the LCTech system FREESTYLE.
For this application the FREESTYLE is equipped with a SPE module.

Put your samples and the clean-up columns DONeX in the racks of the FREESTYLE and hook them in the system. Chose the method in the software or change it dependent on your requirements and start the processing. Now the FREESTYLE system operates your samples reliable around the clock.

FREESTYLE with SPE, EVAporation and GPC Module

Reliable, precise and around the clock: FREESTYLE

Manual Processing →

Protocol of Manual Processing

Extract 10 g of a thoroughly homogenized oat flakes with 50 mL of the extraction solution (acetonitrile/water, 84/16, v/v) in a blender jar at high speed, e.g. with an Ultraturrax.

Pass the extract through a plaited filter. Apply 20 mL representing 4 gram matrix equivalents on the DoneX column using a vacuum manifold. Keep the flow-through for further analysis as it contains the toxin.

Wash the sample reservoir with 10 mL acetonitrile/water (84/16, v/v) and apply the washing solution on the column. Pool the flow-through with the first sample and mix the solutions homogeneously. Evaporate then 7.5 mL of the sample volume (representing 1 gram matrix equivalents) to dryness under nitrogen and reconstitute it in 0.5 mL HPLC solvent.

HPLC Conditions

Deoxynivalenol

Column oven:	33 °C
Separation column:	RP C18 (P/N 10544)
Flow rate:	1 mL/min
Eluent:	90 % 0.01 M acetic acid, 10% acetonitrile
Fluorescence detection with derivatisation:	Post-column derivatisation of Pickerung
	Pinnacle PCX Dual Pump
Column oven:	35 °C
Reactor volume:	Special reactor
Reactor temperature:	115 °C
Reagent 1:	0.15 N NaOH
Reagent 2:	2 M ammonium acetate, 0.03 M methyl acetoacetate
Reagent flow:	0.3 mL/min
Excitation wavelength:	360 nm
Emission wavelength:	470 nm

Recovery Rates

Content of Deoxynivalenol in Oat Flakes	
Deoxynivalenol	
Standard*	100
Recovery rate** Oat flakes 1 ppm Deoxynivalenol	100

** Standard is set = 100 % , ** corrected with non-spiked sample*

Clean-up column DONeX

Chromatograms →

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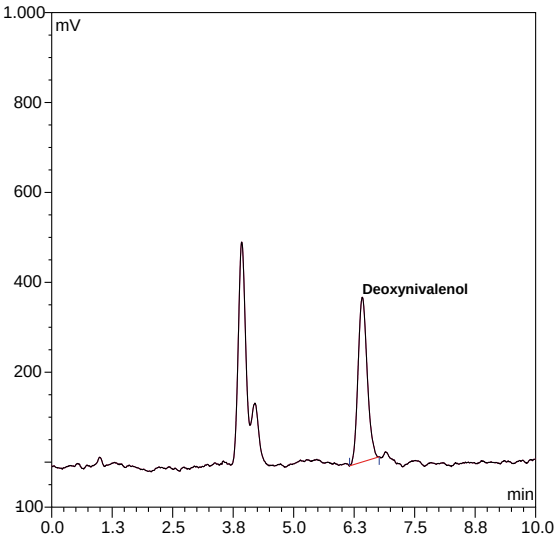
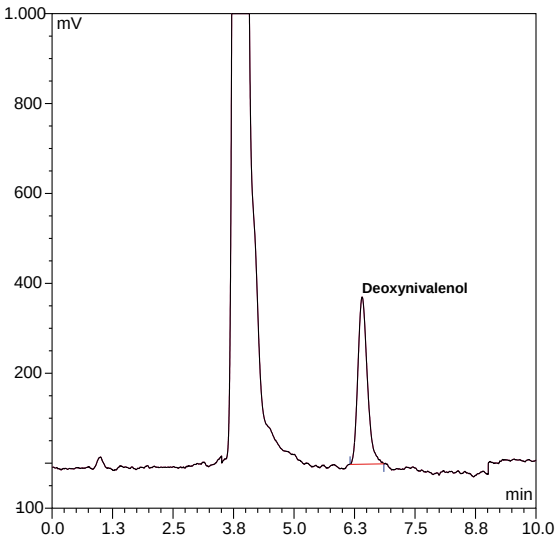
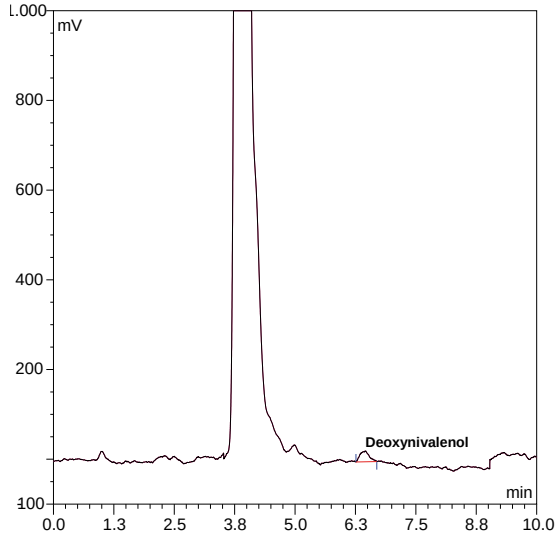
Chromatograms



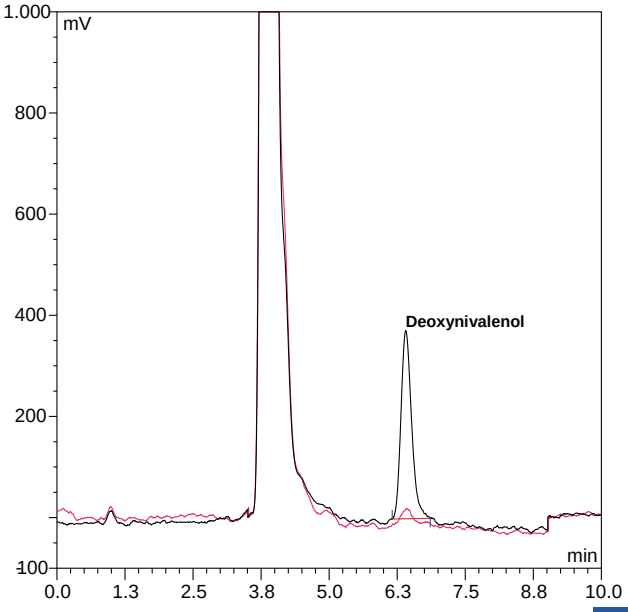
Standard, represents 1 ppm, injection volume 20 µL



Oat flakes, spiked prior to extraction with 1 ppm (1 µg/g) deoxynivalenol



Oat flakes, not spiked, blind sample



Overlay of chromatograms:
blind sample (red)
spiked sample (black)

These LCTech products were used:

DONeX clean-up column,
for DON-analysis
P/N 12792 / 12793

Separation column,
RP C18
P/N 10544

Pickering
Pinnacle PCX Dual Pump
P/N 1153-1072

EluVac
Vaccum Manifold
P/N 12415

FREESTYLE SPE
Robotic system
for sample preparation
P/N 12663 / 12688

Do you have further questions?
Please just write an e-mail to info@LCTech.de!

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