



August 2018

## Deoxynivalenol in Grain ~ 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: [mycotoxins@LCTech.de](mailto:mycotoxins@LCTech.de)

### Sample Preparation

### MYCOTOXINS

#### Deoxynivalenol

Deoxynivalenol (DON) is a worldwide naturally occurring mycotoxin, which is toxic for humans and animals. The toxin is produced by fungi of the fusarium genus e.g. *fusarium culmorum* and *fusarium graminearum*, which are particularly abundant in various cereal crops (wheat, barley, oats).

In agriculture, especially livestock farming, DON impact is as an economic factor, as the toxin causes considerable productivity losses due to deterioration in animal health. For example, a weight gain decrease by 26 % was observed in pigs that ingested DON through their feed.

#### SPE Clean-Up Columns for Analysis of Deoxynivalenol



This toxin is most analysed with an HPLC / UV detector or LC/MS, especially for confirmation analysis. In all processes, thorough sample preparation increases the life time of the analytical system and also the life time of the HPLC system. In addition, pre-cleaning reduces interferences by matrix removal and nearly halves the chromatography time.

For clean-up of deoxynivalenol in food and feed samples, LCTech developed the SPE columns DONeX. They exclude above described interferences through matrix and associated long chromatographies. The DONeX columns are suitable for many common matrices such as corn, barley, oats, wheat, rye, cereal-based feed, but also for more complex matrices such as müsli, pasta or breads. On the following pages, we examined some grain products for you.

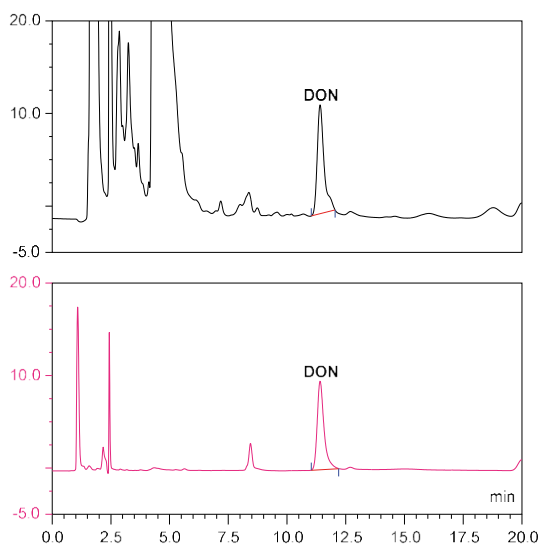
The clean-up column is available in a 3 mL format and is thus suitable for manual as well as for automated processing around the clock with the LCTech robotic system FREESTYLE SPE.

## Processing Protocol

Homogenise 10 g of durum wheat/wheat flour/chicken feed and extract it with 50 mL acetonitrile/water (84/16 (v/v)) in a beaker at high-speed level, e.g. with an Ultraturrax. Filtrate the extract with a folded filter. Load 20 mL (represents 4 g matrix) using a vacuum manifold (e.g. LCTech EluVac) onto the DONeX column. Wash the column with 10 mL of acetonitrile/water (84/16 (v/v)). The flowthrough- and the washing-fraction are combined.

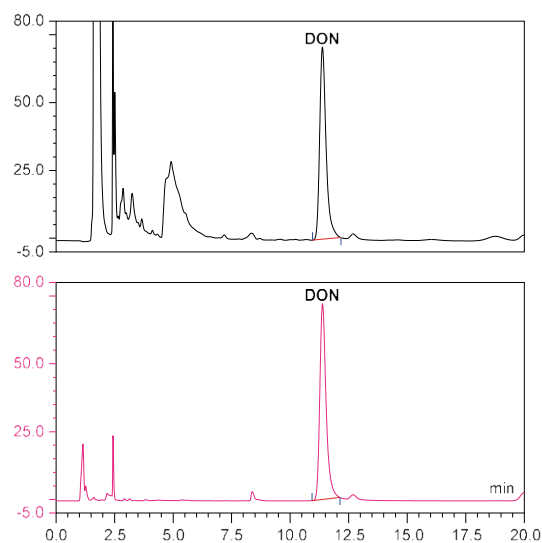
Evaporate 7.5 mL of the mixed solution (represents 1 g matrix) with nitrogen until dryness and dissolve it afterwards in 0.5 mL of HPLC-solvent.

## Chromatograms Durum Wheat



Red: Standard 1,5 ppm

Black: Durum Wheat 1,5 ppm



Red: Standard 5 ppm

Black: Durum Wheat 5 ppm

## HPLC-Conditions (Deoxynivalenol)

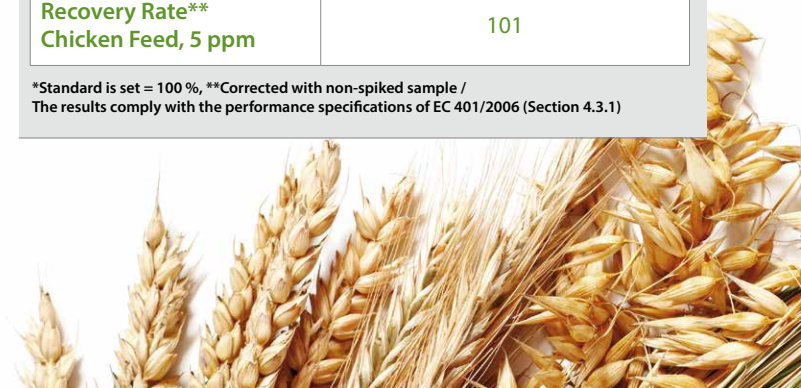
Mycotoxin:	Deoxynivalenol (DON)
Column Oven:	35 °C
Separation Column:	RP C-18 (P/N 10522)
Flow Rate:	1 mL/min
Eluent:	95 % HPLC-water, 5 % acetonitrile
UV Detection:	218 nm

## Recovery Rates

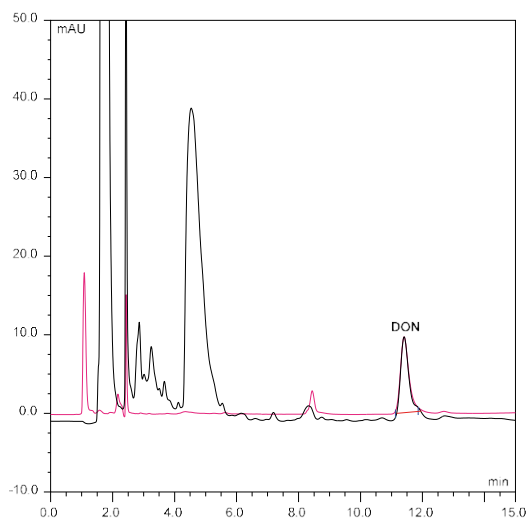
Content of Deoxynivalenol in  
Semolina/Wheat Flour/Chicken Feed

Mycotoxin	Deoxynivalenol (DON)
Standard*	100
Recovery Rate** Durum Wheat, 1,5 ppm	101
Recovery Rate** Durum Wheat, 5 ppm	99
Recovery Rate** Wheat Flour, 1,5 ppm	98
Recovery Rate**** Wheat Flour, 5 ppm	99
Recovery Rate** Chicken Feed, 1,5 ppm	104
Recovery Rate** Chicken Feed, 5 ppm	101

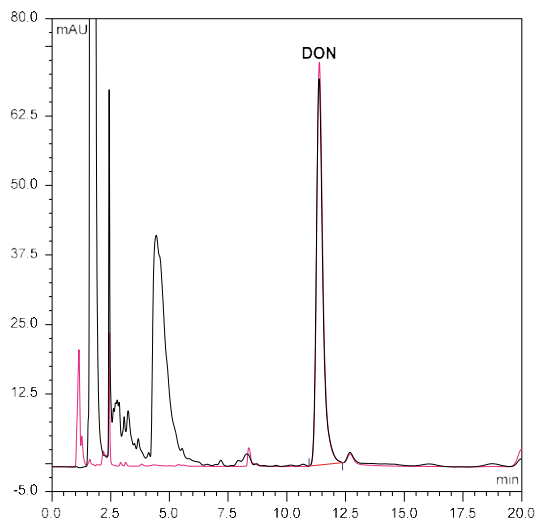
\*Standard is set = 100 %, \*\*Corrected with non-spiked sample /  
The results comply with the performance specifications of EC 401/2006 (Section 4.3.1)



## Chromatograms Wheat Flour

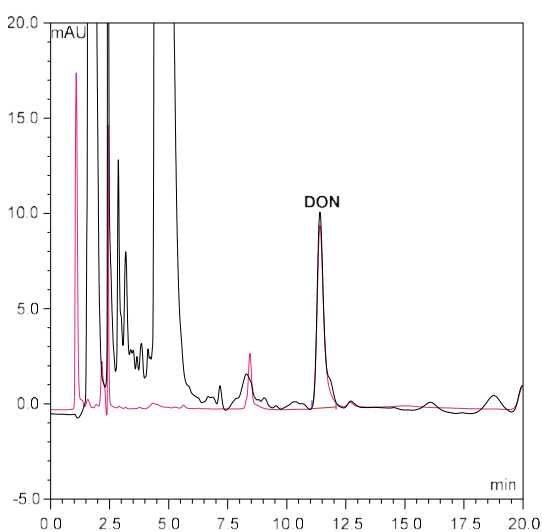


*Red: Standard 1,5 ppm*  
*Black: Wheat Flour 1,5 ppm*

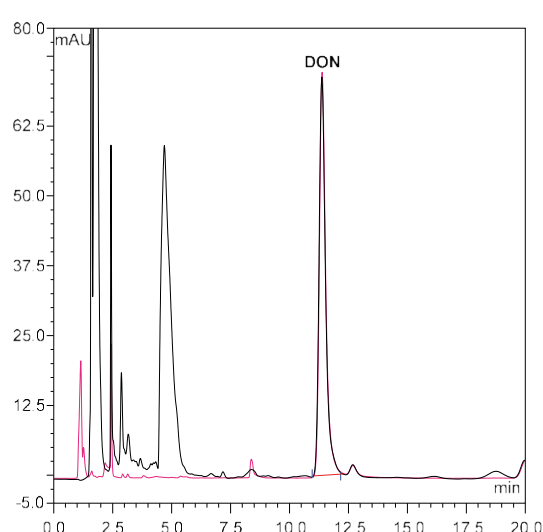


*Red: Standard 5 ppm*  
*Black: Wheat Flour 5 ppm*

## Chromatograms Chicken Feed



*Red: Standard 1,5 ppm*  
*Black: Chicken Feed 1,5 ppm*



*Red: Standard 5 ppm*  
*Black: Chicken Feed 5 ppm*

## Conclusion

As you can see in the chromatograms, using the LCTech DONeX clean-up column, long chromatographies and matrix interferences can be reliably excluded. This results in a better, and faster chromatography as well as a higher sensitivity of the measurement.

In addition, especially in combination with automation you achieve a very high sample throughput and reproducible results with the clean-up columns.

## These LCTech products were used:

DONeX, SPE Columns for DON-Analysis  
P/N 12792 / 12793

HPLC Separation Column RP C-18  
P/N 10522

EluVac Vacuum Manifold  
P/N 12415

FREESTYLE SPE Robotic System for Automated  
Sample Preparation  
P/N 12663, 12668