



December 2020

Deoxynivalenol in Christmas Biscuits ~ 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

Sample Preparation

MYCOTOXINS

Spiced Christmas Biscuit and Cinnamon Stars

The pre-Christmas season has started. At every corner there is a fine fragrance of biscuits, gingerbread and mulled wine. The time of the year to feast and enjoy. Biscuit baking was probably developed in the 18th century and there are already more than 20,000 different varieties of Christmas biscuits. Traditional sweets at Christmas time include Spiced Christmas Biscuit and Cinnamon Stars. Both biscuits are very popular in Germany at this time.

Spiced Christmas Biscuit is a flat shaped biscuit made of a spiced shortcrust. The pastry seems to have originated in Belgium or the Netherlands. Cinnamon Stars are a Christmas Biscuit made from egg whites, sugar, almonds, cinnamon and flour, and originate from Swabia. Both, flour and cinnamon can be contaminated with mycotoxins. For this reason, cinnamon and flour are regulated by a maximum level of mycotoxins which they must not exceed on import.

Deoxynivalenol Monitoring Made Easy

Deoxynivalenol is a metabolic product of various fungi of the genus *Fusarium* found in cereals, flours and pasta. Normally the toxin is analysed by HPLC with UV detector or by LC/MS or alternatively by HPLC with post-column derivatisation and subsequent fluorescence measurement.

Good sample preparation is important for all three methods. LCTech supports you in your sample preparation workflow with the DONeX clean-up column. The column developed by LCTech excludes interferences by the matrix and associated long chromatographies and disturbing matrix peaks. Better, faster chromatograms and higher measurement sensitivity, less contaminated analytical equipment and higher measurement specificity are the results.



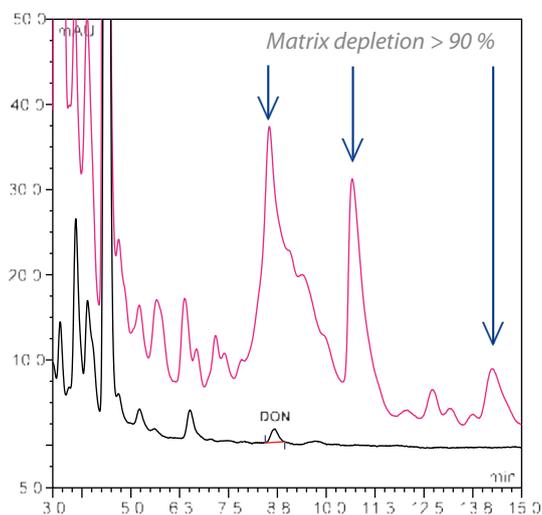
DONeX™

Processing Protocol

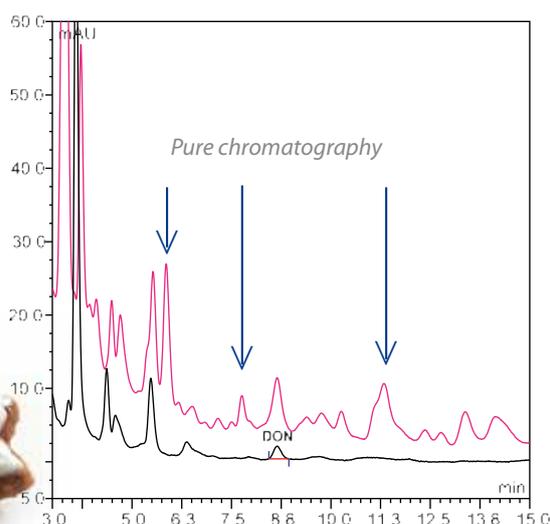
Extract 10 g homogenised Spiced Christmas Biscuit or Cinnamon Stars with 50 mL acetonitrile/water (84/16 (v/v)). To ensure high extraction efficiencies, continue the extraction for at least 10 - 15 minutes. Then you can centrifuge the extract at 3000 x g for 5 minutes or you can filter it through a pleated filter. Load 20 mL of the extract (equivalent to 4 g) onto a DONeX clean-up column.

Collect the flow through in a 50 mL polypropylene tube. Rinse the sample tube with 10 mL acetonitrile/water (84/16 (v/v)). Load this solution onto the column and collect the flow through with the 50 mL polypropylene tube. Mix the solutions. Fill 7.5 mL (equivalent to 1 g) into a glass tube and evaporate it to dryness under a nitrogen stream. Add 500 µL HPLC solvent or HPLC water and mix vigorously to dissolve the analytcs. Filter the sample through a 0.2 µ syringe filter into a GC vial. Now you can analyse the sample with the appropriate standards by HPLC-UV.

Chromatograms



Black: Spiced Christmas Biscuit (500 ppb) cleaned with DONeX
Red: Spiced Christmas Biscuit without clean-up



Black: Cinnamon Stars (500 ppb) cleaned with DONeX
Red: Cinnamon Stars without clean-up



➔ The DONeX columns also allow the clean-up of modified forms of deoxynivalenol and nivalenol. High matrix load - up to 4 g.

HPLC-Conditions

Deoxynivalenol

HPLC:	isocratic
Column Oven:	33 °C
Separation Column:	RP C-18 (P/N 10522)
Flow Rate/ Eluent:	1.0 mL/min; HPLC-water/acetonitrile/ acetic acid (95/5/1 (v/v/v))
Absorption Wave- length:	215 nm

Recovery Rates

Content of Deoxynivalenol in Spiced Christmas Biscuit and Cinnamon Stars

Mycotoxin	Deoxynivalenol
Standard*	100
Recovery Rate** Spiced Christmas Biscuit with DONeX	100
Recovery Rate** Cinnamon Stars with DONeX	106

* Standard was set = 100% , ** Corrected with non-spiked sample / The results are in accordance with the performance specifications of the EC 401 / 2006 (section 4.3.1).

These LCTech Products were used:

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

HPLC Separation Column RP C-18
P/N 10522

EluVac Vacuum Manifold
P/N 12415