Mycotoxins in black sesame seeds

Cleaned-up with *CrossTOX®*

Sesame

Aromatic, nutty, fine: sesame seeds are tiny vitamin gobs and a valuable contribution to a balanced diet. Often misjudged as a “garnish“ on bread rolls, sesame seeds offer many more possibilities: Freshly roasted sesame seeds refine vegetable, fish and meat dishes and give salads that special taste. Black sesame is considered the original form of sesame and is particularly rich in health-promoting ingredients. The varieties also differ in taste: black sesame is spicier and nuttier than white sesame.

Currently, several sesame and sesame-containing products in different varieties are affected by a product recall. The reason is residues of ethylene oxide in the sesame seeds used. The chemical is toxic and carcinogenic.

At increasing doses, it can even lead to convulsions or coma, according to the notice of the consumer portal produktrückrufe.de.

18 Mycotoxins at a stroke - CrossTOX® makes it possible

LCTech’s CrossTOX® columns enable highly efficient sample clean-up of regulated and expected mycotoxins. At the same time, they improve the conventional dilute-and-shoot application by a QuEChERS-based procedure.

A specially adjusted sorbent guarantees high depletion of analytically interfering substances even with difficult matrices. CrossTOX® can process grain-based matrices as well as nuts, dried fruits and spices with very good results. The loading capacity is 3 mL (corresponds to 0.6 g matrix).

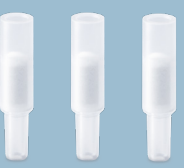
Clean-up via CrossTOX® is either manually or automated possible with a FREESTYLE SPE robotic system. Full automation can be achieved: Just combine it with an HPLC direct injection module. Depending on the matrix, the majority of analytes are measured without internal standards and with excellent recoveries. Due to the reduction of internal standards and the purity of the sample, enormous costs per sample as well as maintenance costs (LC-MS/MS) are saved.

These LCTech products were used:

17900 CrossTOX® 100 pcs/box (manual)

17901 CrossTOX® 100 pcs/box (automated)

More sample measurements in less time with clean-up columns ***CrossTOX®*** from LCTech!



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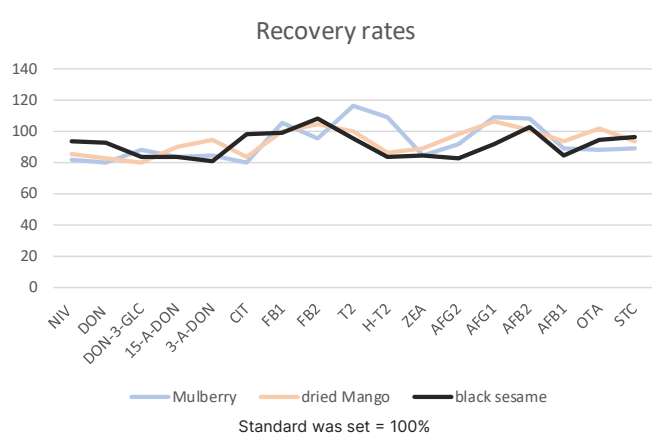
Processing Protocol

Extract 10 grams of homogenised black sesame with 50 mL acetonitrile/water (84/15 (v/v) + 1% acetic acid). Carry out the extraction for at least 5-30 minutes to gain high extraction efficiency.

Filter the crude extract or centrifuge it for 5 min. Load max. 3mL of the clear supernatant onto a CrossTOX® column with a flow rate of 1-2 mL/min and collect the flowthrough with a sample vial for analysis by LC-MS/MS. For more efficient matrix reduction, reduce the sample volumes down to 500 µL instead of 3mL.



Only for the analysis of black sesame a correction by means of internal standards for the analytes T2/H-T2, zearalenone and aflatoxin G2 was necessary(*). The remaining analytes could be analysed by means of an external calibration; no addition of internal standards is required for quantification, as the analyte concentrations were not affected by the clean-up using the CrossTOX® columns and their efficient matrix depletion.

Conclusion

The recoveries for the mentioned analytes are between 80 and 120 % and thus meet the requirements of EN401/2006. As a comparison matrix, dried mango and mulberries were processed with the same protocol to demonstrate the matrix compatibility of the CrossTOX® column.

Spiking concentration	
Analyt	**Concentration (µg/ kg)**
Aflatoxin B1/G1	8
Aflatoxin B2/G2	2
Ochratoxin A	20
Zearalenone	100
FB1/FB2	250
T2/H-T2	50
Sterigmatocystin	50
Dexynivalenol	400
Dexynivalenol 3-Glykoside	400
15-Acetyl-Deoxynivalenol	400
3-Acetyl-Deoxynivalenol	400
Nivalenol	400
Citrinin	25

Conditions	
UPLC	gradient
Column oven	38 °C
Separation column	Accure Biphenyl 100mm x 2.1mm; 2.6µm with precolumn
Flowrate, LC solvent	0.4 mL/min; LC solvent A: HPLC water/methanol (98/2 (v/v), 5mm ammonium-acetate, 1% acetic acid) LC solvent B: HPLC water/ methanol (2/98 (v/v), 5mm ammoniumacetate, 1% acetic acid)
0 - 2 min	95% A; 5% B
2 - 5 min	15% A; 85% B
5 - 11 min	5% A; 95% B
11 - 16 min	95% A; 5% B
Analytics	Heated ESI 3500 V (+); 1500 V (-); Ion-Transfer-Tube 325°C; Verdampfer 350°C

Do you have a special request as to which matrix we should test for you?
Contact us by e-mail at: info@LCTech.de

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LCTech GmbH
Daimlerstr. 4
84419 Obertaufkirchen, Germany
Tel. +49 8082 2717-0
info@LCTech.de
www.LCTech.de