Food Standards Scotland

For safe food and healthy eating

Oat-Based Commodity and Non-Dairy Alternative Drinks Chemical Contaminants Survey

Report by Fera Science Ltd.

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Report of Oat-Based Commodity and Non-Dairy Alternative Drinks Chemical Contaminants Survey for Food Standards Scotland
Fera Science Ltd.

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2. Glossary

AFB1	Aflatoxin B1
AFB2	Aflatoxin B2
AFG1	Aflatoxin G1
AFG2	Aflatoxin G2
DAS	Diacetoxyscirpenol
DON	Deoxynivalenol
DON-3-G	Deoxynivalenol 3-glucoside
FB1	Fumonisin B1
FB2	Fumonisin B2
FB3	Fumonisin B3
FSS	Food Standards Scotland
FUSX	Fusarenon X
GB	Great Britain
GE	Glycidyl esters
HPLC-UV	High Performance Liquid Chromatography with Ultra Violet Detection
HT-2	HT-2 toxin
IAC	Immunoaffinity Column
LC-MS/MS	Liquid Chromatography Tandem Mass Spectrometry
LOQ	Limit of Quantification
3-MCPD	3-Monochloropropane diol
MU	Measurement Uncertainty
NEO	Neosolaniol
NIV	Nivalenol
OTA	Ochratoxin A
PBS	Phosphate Buffered Saline
RASFF	Rapid Alert System for Food and Feed
STG	Sterigmatocystin
T-2	T-2 toxin
T2-3α-G	T-2 toxin 3α-glucoside
α, β-ZOL	α, β-Zearalenol
ZON	Zearalenone

3. Executive Summary

Food Standards Scotland (FSS) commissioned a study with Fera Science Ltd. to carry out a survey of the potential mycotoxin content in oats and high oat content cereal products, oat drinks, soya based infant formula and soya drinks, almond drinks and coconut drinks. The survey requirement was to test for *Fusarium* mycotoxins, including some modified forms, aflatoxins, ochratoxin A and fumonisins in the oat products and soy products. Modified mycotoxins, are structurally modified as an effect of a metabolic process exerted by a living organism (i.e. plants, fungi, mammals), or as an effect of food processing (EFSA definition). Some plants, including cereals, are able to metabolise mycotoxins into more polar metabolites as a part of the plants defence. For almond and coconut drinks only aflatoxins and ochratoxin A were required. In addition, isoflavone analysis was requested for the soy products and 3-MCPD and glycidyl esters were required for the coconut drinks.

Forty one products were purchased from a range of retail and online sellers from Scotland. Samples were based on the following categories: oats and high oat content cereal products, oat drinks, soya based infant formula and soya drinks, almond drinks and coconut drinks following a sampling plan designed by FSS. As far as possible, oat products made from Scottish oats were selected for sampling. Two methods were used to analyse the samples to give results for all the requested mycotoxins. Some additional mycotoxins were included in the analysis.

Mycotoxins were detected in several oat products, although no sample exceeded any maximum level (ML) as established in The Contaminants in Food (Scotland) Regulations 2013 and Retained Commission Regulation (EC) No 1881/2006 (1, 2, 3) or the Indicative Levels in Commission Recommendation 2013/165/EU on the presence of T-2 and HT-2 toxin in cereals and cereal products (4). Eleven samples contained DON levels from 6.1 to 233 µg/kg. The sample with the highest DON level (porridge oats), also contained the highest 3-AcDON (37.5 µg/kg), HT-2 (45.2 µg/kg), and T-2 (10.9 µg/kg) levels and a low level of T-2-3α-G. Eight samples contained HT-2 above the LOQ (levels from 7.5 to 45.2 µg/kg) and three contained T-2 (8.5 to 10.9 µg/kg).

DON-3-G was found in five samples, the highest level was found in the sample that contained the highest DON level. Two samples contained ZON, no modified forms of ZON were detected in any samples. It was not possible to analyse for modified HT-2 as there is

no commercially available source of an analytical standard for this compound and it was not possible to obtain it from any other source.

Mycotoxins were also detected in the non-dairy alternative drinks at levels below MLs in place for other cereal products. There are no MLs in force for these products. The highest mycotoxin levels in non-dairy alternative drinks were for DON in four oat drinks, these samples also contained DON-3-G and the highest HT-2 and T-2 levels. The sample with the highest DON, HT-2 and T-2 levels also contained T-2-3α-G above the LOQ. Low levels of aflatoxin B1 were detected in some samples, one soya drink also contained DON, and fumonisin B1 as well as a trace level of ZON.

Mycotoxins were frequently observed just below the current LOQ (the lowest level the method was validated for) in the drinks samples. If required, for example for consumer intake studies, this LOQ could be reduced further. More work may be required to improve the current multi-mycotoxin analytical method to obtain lower limits of quantification for some compounds e.g. nivalenol for the drinks samples.

No 3-MCPD or glycidyl esters were detected in the five coconut samples analysed.

Isoflavones were measured in soya products; total isoflavone content ranged from 213 to 488 mg/L (mg/kg for infant formula). These are no maximum levels set for these compounds that occur naturally in soy products.

This study has shown the potential for the co-occurrence of mycotoxins in oat products and non-dairy alternative drinks. Modified forms of mycotoxins were detected. Deoxynivalenol-3-glucoside (DON3G), a conjugate form of deoxynivalenol (DON) and T-2 α3-glucoside, a conjugated form of T-2 toxin, were detected, as well as 3-AcDON and 15-AcDON, the acetyl derivatives of DON that are produced by *Fusarium* species.

5.1 Background to the study – oat products

In 2017 the European Food Safety Authority (EFSA) established a tolerable daily intake (TDI) for T-2 and HT-2 of 0.02 µg/kg body weight (bw) per day based on a new toxicity study in rats that confirmed that immune- and haematotoxicity are the critical effects of T-2 (5). Fusarium mycotoxins, particularly T-2/HT-2, are of concern in oat products due to a current proposal for amendments to European Commission (EC) regulations on Maximum Levels (MLs) in cereals and cereal grain products. There are currently no maximum levels in place for T-2 and HT-2 toxins, although a Commission Recommendation in 2013 introduced Indicative Levels above which an investigation should be performed (4). There have been ongoing discussions within the European Commission for a number of years about introducing maximum levels for T-2 and HT-2, (6) and suggested maximum levels are under discussion (7). The maximum levels proposed are lower than the Indicative Levels and are a reflection of the lower TDI established in 2017 by EFSA (5). The proposed maximum level is 10 µg/kg for sum T-2 and HT-2 in infant food compared to 15 µg/kg in Recommendation 2013/165/EU, while it is 600 µg/kg in oats (with husk) compared to 1000 µg/kg in the recommendation (4). Cereal products such as biscuits, snacks and breakfast cereals have a proposed limit of 20 µg/kg, but indicative levels ranged from 25 to 75 µg/kg for these products.

Of the *Fusarium* mycotoxins, T-2/HT-2 production is linked to climates with high moisture conditions during crop production and storage, as such, it is more prevalent in Northern European countries such as Scandinavian countries, UK and Ireland (8, 9). Previous surveys of T-2/HT-2 in UK oats and oat products found that several products, particularly those of Scottish origin, would be at or exceed the new proposed regulatory limits during a year of especially high T-2/HT-2 levels in unprocessed grain (10).
Other mycotoxins such as aflatoxins and ochratoxin A and other *Fusarium* mycotoxins (deoxynivalenol and zearalenone) are subject to maximum levels that are in force from retained European legislation (1, 2, 3).

In addition to this, an emerging risk of modified mycotoxins has been identified (11).
Testing capacity for these compounds have proved to be a challenge due to the lack of available analytical standards and reference materials resulting in a lack of validated

methods and, as such, most available data on mycotoxin content in cereals is only based on unmodified forms. Modified mycotoxins are not currently included in regulation maximum levels, however, there is evidence that they may contribute to the overall health risk from mycotoxins exposure as they can be converted to the parent, more bioavailable, forms of the mycotoxins in the gut (12, 13).

5.2 Background to the study – non-dairy alternatives

There has been an increase across GB of consumption of non-dairy alternative products. These include oat, almond, soya and coconut-based products particularly in the form of drinks, but also other products such as yoghurt alternatives. Many of these are produced from ingredients known to be at risk from mycotoxin contamination. For example, almond and coconut products may contain mycotoxins in the form of aflatoxin and ochratoxin A, oats are known to be at risk of containing *Fusarium* mycotoxins, as are soya products. The observations relating to modified mycotoxins detailed in 5.1 apply equally to dairy alternatives.

Maximum levels have been set for 3-MCPD in soya sauce and hydrolysed vegetable protein and for glycidyl esters (GE) vegetable oils and infant formula as there is some concern that infants receiving only formula may slightly exceed the safe level for these compounds (1, 2, 3). There have been reports through the Rapid Alert System for Food and Feed Portal (RASFF) of coconut products containing 3-monochloropopane diol (3-MCPD) and glycidyl esters, therefore it was important to obtain information on their occurrence in products that may be consumed by children.

Soya-based products are also a potential source of phytoestrogens in the form of isoflavones. The UK Committee on Toxicology produced a statement on the risks of high isoflavone consumption by infants (14). The main toxicological concern from infants' consumption was the potential of these compounds to disrupt the development of the reproductive system. Other possible adverse effects related to the immune system and thyroid function (14). Therefore, it was desirable to obtain data on the concentrations of isoflavones in soya drinks and infant formula.

5.3 Aims and Objectives of the Study

The aim of this project was to undertake a survey to address the evidence gap on levels of modified and unmodified *Fusarium* mycotoxins [including T-2/HT-2, Deoxynivalenol (DON), Nivalenol (NIV), Zearalenone (ZON)], Fumonisins B1, B2, B3, and Aflatoxins B1, B2, G1, G2, and Ochratoxin A (OTA), in a range of non-dairy alternative drinks, oat-based cereal, biscuit and oatcake products of Scottish origin. An additional aim was to obtain information on 3-MCPD non-dairy alternative coconut drinks and isoflavones in soya drinks and soya-based infant formula.

6. Methodology

6.1 Samples

Sample purchase and collection was subcontracted to HallMark Veterinary & Compliance Services. FSS designed the sampling plan and provided a detailed list of suggested products, this was used to plan purchase and collection of samples. This list was designed to give sampling from retailers including national and local suppliers with limited stockists across Scotland. The sampling plan requested 40 samples, but an extra sample of oatmeal purchased in error instead of oatcakes was also included resulting in a total of forty-one samples that were included in the survey.

Samples were purchased in September 2021 with the intention that all products would have been manufactured using the previous years' harvest, i.e. were not from 2021 crop. A small number of samples (4 out 41) were purchased from on-line retailers. In all cases three retail packs from one batch of each product were purchased resulting in sample sizes from 450 g to 3000 g (or 3000 ml for liquids). A full detailed list of the samples collected has been provided separately, and a summarised list is given in Annex A (Table 5). This was a limited survey that only included small numbers of each type of product and as such was not fully representative of the market, therefore it was not appropriate to name brands.

6.2 Sample preparation and storage

On receipt, samples were immediately logged into the laboratory information management system (LIMS) and given a unique identifying number. Storage conditions on the package were adhered to and any samples with short expiry dates, e.g. some drinks, were transferred to plastic bottles and stored in the freezer until analysis according to standard practice and in-house quality procedures to preserve sample integrity and avoid any changes to potential contaminant levels in the products.

For solid oat products (porridge, biscuits, oatcakes), for each sample all three sample packs received were combined, and for biscuits and oatcakes the products were roughly broken up by hand. Samples were milled or cryomilled to pass through a 1 mm mesh, to produce a finely ground sample. Each sample was mixed for 30 minutes after milling to ensure homogeneity. All milling and mixing equipment was thoroughly cleaned between each

sample to prevent cross contamination. All samples were stored in the freezer after grinding and homogenisation. For drinks, samples were shaken well to mix before aliquots were removed for analysis. All samples were stored in the freezer after opening.

6.3 Sample analyses

Samples were divided into product categories with specific analytical requests for each product. These had been specified by FSS and are set out in Table 1. The analytes requested included Type A trichothecene mycotoxins, e.g. T-2 toxin, HT-2 toxin, Type B trichothecenes (deoxynivalenol, nivalenol), as well as other *Fusarium* mycotoxins (zearalenone, fumonisins), and other mycotoxins of significant health concern (aflatoxins and ochratoxin A). Modified forms of trichothecenes were also requested, as well as processing contaminants (3-MCPD and glycidyl esters) in coconut products and isoflavones in soya products.

Table 1. Product categories and requested analyses

Product	**Testing Requirements**	**Number of samples purchased**
Oats and high oat content products	Unmodified mycotoxins (T-2/HT-2, DON, NIV, ZON, Fumonisins B1, B2 and B3, Aflatoxins B1, B2, G1, G2, OTA) Modified mycotoxins (DON and T-2/HT-2)	17
Oat drinks	Unmodified mycotoxins (T-2/HT-2, DON, NIV, ZON, Fumonisins B1, B2 and B3, Aflatoxins B1, B2, G1, G2, OTA) Modified mycotoxins DON and T-2/HT-2	4
Soya drink	Unmodified mycotoxins (T-2/HT-2, DON, NIV, ZON, Fumonisins B1, B2 and B3, Aflatoxins B1, B2, G1, G2, OTA) Modified mycotoxins DON and T-2/HT-2 Isoflavone glycosides (genistin, daidzin, and glycitin), aglycones (genistein, daidzein, and glycitein) and total isoflavones	8
Soya-based Infant Formula	Unmodified mycotoxins (T-2/HT-2, DON, NIV, ZON, Fumonisins B1, B2 and B3, Aflatoxins B1, B2, G1, G2, OTA) Modified mycotoxins DON and T-2/HT-2	2

	Isoflavone glycosides (genistin, daidzin, and glycitin) and aglycones (genistein, daidzein, and glycitein)	
Coconut drink	Unmodified mycotoxins (Aflatoxins B1, B2, G1, G2, OTA) 3-MCPD and glycidyl esters	5
Almond drink	Unmodified mycotoxins (Aflatoxins B1, B2, G1, G2, OTA)	5

For practical and logistical purposes in the laboratory; soya products and all oat products and alternative non-dairy drinks were analysed by two analytical methods giving full coverage of all mycotoxins requested by FSS, with the exception of modified HT-2 toxin. This analyte could not be included as an analytical reference standard was not commercially available at the time of the study.

Fera has ISO17025 accreditation for several mycotoxin methods including a method for a suite of 17 *Fusarium* mycotoxins, including modified forms, by LC-MS/MS; a method for ten trichothecenes; as well as aflatoxins B1, B2, G1, G2 and OTA in a range of matrices, including cereals and cereal products using immunoaffinity column clean-up and HPLC with fluorescence detection. Mycotoxin methods are required to meet the performance characteristics given in Retained Commission Regulation (EC) No 401/2006 as a minimum (15). The general operation of LC-MS methods is accredited and Fera has Flexible Scope accreditation that allows accreditation to be claimed for certain analyte, matrix and instrument combinations. Some results in the study are not accredited, but all work was undertaken to ISO 17025 quality standards.

6.4 Mycotoxin Analysis using 11+ Immunoaffinity column clean-up

All samples were analysed for mycotoxins using an in-house method for the determination of 11+ mycotoxins by immunoaffinity column (IAC) clean-up and LC-MS/MS analysis. This method is not yet accredited. The method uses immunoaffinity columns with antibodies specific to certain mycotoxins and has claimed performance for the following mycotoxins:

- Aflatoxins B1, B2, G1 and G2
- Ochratoxin A (OTA)
- Deoxynivalenol (DON)
- T-2 and HT-2 toxin

- Fumonisins B1 and B2
- Zearalenone

In addition, due to some cross reactivity of the antibodies the columns also cross-react to varying degrees with:

- 3-Acetyldeoxynivalenol
- T-2 toxin α-glucoside
- Fumonisin B3
- α-zearalenol and β-zearalenol
- Sterigmatocystin
- Deoxynivalenol 3-glucoside

This allowed some analytes to be included in the survey that had not been specifically requested (i.e. 3-acetyldeoxynivalenol, α-zearalenol, β-zearalenol and sterigmatocystin).

The method had previously been validated in-house for oat products for all of the above analytes. DON-3-G had not produced satisfactory data due to low recovery rates, which was shown to be due to low cross reactivity of DON-3-G to the DON antibody in the IAC. The method was able to detect DON-3-G in test samples, however the results were indicative due to low recovery. The accredited method (Suite of 17 *Fusarium* mycotoxins) was used to quantify the mycotoxins that could not be analysed using this method. The 11+ IAC method will undergo interlaboratory validation in 2022 through an independent laboratory validation scheme.

6.4.1 Oat products – mycotoxins analysis by 11+ method

For solid oat products, aliquots of sample (25 g) were extracted by blending at high speed for 2 minutes with 70% methanol. Following this, samples were filtered or centrifuged, and an aliquot (20 mL) of supernatant (or filtrate) was diluted with Phosphate Buffered Saline (PBS). An aliquot of the diluted extract was cleaned up by passing through the IAC under gravity. The IAC was washed with water, then dried by passing air through before the analytes were eluted by passing methanol, then water though the IAC into a vial. This was mixed well, and if necessary, filtered through a syringe filter before being transferred to an autosampler vial for LC-MS/MS analysis.

Quality control samples including procedural blanks, in-house reference samples, and spiked samples, were included in the analytical batch to check accuracy (recovery and comparison to assigned values), and blank control, i.e. no contribution from reagents or laboratory environment. Limits of quantification (LOQ) for the 11+ method are given in Table 2 (below). In this case LOQ is defined as the lowest level validation has been undertaken, not as the lowest level that can be quantified.

6.4.2 Drinks and infant formula – analysis by 11+ method

For liquid drinks, 25 g of sample was diluted with 62.5 mL of PBS. This was mixed by shaking then centrifuged. The supernatant was filtered and an aliquot (30 mL) cleaned up on IAC using the same procedure as for the oat products. Samples were analysed by LC-MS/MS using a different calibration range than oat products.

For the 2 infant formula samples, a portion of sample (25 g) was weighed. This was dissolved in 250 mL of water, using an ultra-sonic bath to aid dispersion / dissolution. An aliquot (25 mL) of reconstituted formula was analysed in the same manner as the liquid samples.

Table 2. Limits of Quantification (LOQ, lowest validated level) for analytes in 11+ method

Compound	**LOQ Oat products (µg/kg)**	**LOQ Non-dairy alternative milks (µg/kg)**
Required compounds		
Aflatoxin B1	0.25	0.01
Aflatoxin B2	0.25	0.01
Aflatoxin G1	0.25	0.01
Aflatoxin G2	0.25	0.01
Ochratoxin A	0.25	0.01
Deoxynivalenol	5	0.2
T-2 toxin	5	0.2
HT-2 toxin	5	0.2

Nivalenol	n/a	n/a
Zearalenone	2.5	0.1
Fumonisin B1	10	0.4
Fumonisin B2	5	0.2
Fumonisin B3	5	0.2
Deoxynivalenol-3-glucoside	5	0.2
T-2 toxin α-3-glucoside	5	0.2
Additional compounds		
3-Acetyldeoxynivalenol	5	0.2
Sterigmatocystin	0.25	0.01
α-zearalenol	2.5	0.1
β-zearalenol	2.5	0.1

6.5 Mycotoxins analysis using In-house method FSG 818 - Multi mycotoxins by LC-MS/MS

Samples were also analysed by a second In-house method (FSG 818) - Method for the extraction and LC-MSMS analysis of 17 mycotoxins, that is accredited to ISO17025 for analysis of cereals and cereal based animal feed. Analytes included in the method and their LOQs are listed in Table 3.

Table 3. LOQs for analytes included in In-house method FSG 818.

Compound	**LOQ / µg/kg**
3-Acetyldeoxynivalenol	10
15-Acetyldeoxynivalenol	20
Deoxynivalenol	10
Deoxynivalenol-3-glucoside	10
Diacetoxyscirpenol	10
Fusarenon X	10
HT-2 toxin	10
Neosolaniol	10
Nivalenol	50
T-2 toxin	10

Compound	LOQ / µg/kg
T-2 toxin-α3-glucoside	10
α-Zearalenol	2.5
β-Zearalenol	2.5
α-Zearalenol-14-glucoside	5
β-Zearalenol-14-glucoside	5
Zearalenone	2.5
Zearalenone-14-glucoside	5

In addition, 13C isotopically labelled standards were used as internal standards to control the analysis. 13C Labelled standards used in the method were:

^{13}C17-3-Acetyldeoxynivalenol

^{13}C15-Deoxynivalenol

^{13}C19-Diacetoxyscirpenol

^{13}C22-HT-2 toxin

^{13}C15-Nivalenol

^{13}C24-T-2 toxin

^{13}C18-Zearalenone

Results for oat products (porridge etc.) using this method are accredited to ISO17025.

6.5.1 Oat products – analysis by accredited multi mycotoxin method - In-house method FSG 818

An aliquot (5 g) of homogenised sample was extracted by shaking with a solvent mixture of water and acetonitrile, (16 : 84, v/v) for 2 hours. Samples were centrifuged then an aliquot of the extract was cleaned up using a solid phase extraction (SPE) cartridge. The cleaned-up extract was then dried, and reconstituted in a solvent suitable for LC-MSMS analysis, internal standards (as detailed above) were added at this point. After separation using LC, detection was by tandem quadrupole MS, using an electrospray source and a combination of positive and negative mode ionisation. Samples were analysed alongside calibration standards, which were used to quantify any residues detected. Identification of the analytes was confirmed by comparison of retention times and peak area ion ratios with those of the calibration standards according to in-house SOP FSG002 rev 11, LCMS

analysis based on internationally recognised parameters (16). All data met the specified quality parameters for retention time an ion ratio unless indicated.

6.5.2 Drinks and infant formula – analysis by accredited multi mycotoxin method - In-house method FSG 818

Aliquots of liquid samples (5 g) were extracted in the same way as the oat products above. Soy infant formula was reconstituted as in section 5.6 and a 5 g aliquot taken for analysis. Results for liquids (non-dairy alternative milks) are not accredited.

6.6 Coconut Drinks – analysis for 3-MCPD and glycidyl esters

Five coconut drink samples were extracted to obtain the fat from the samples, the 3-MCPD, 2-MCPD and glycidyl esters were extracted from the fat obtained from this. Initially a single aliquot (10 g) of each sample was weighed into a tube. Hexane (15 mL) was added and this was shaken and sonicated for 15 minutes each. This was centrifuged for 5 minutes at 4500 rpm, the hexane layer was transferred to a glass vial and extracted with 5 mL hexane by shaking for 15 minutes. This was centrifuged at 4500 rpm for 5 minutes and the supernatant added to the glass vial. The solvent was evaporated under N_2 at 40˚C until an oily residue remained and no hexane left. Diethyl ether was added and the extract transferred to a glass vial. MCPD esters were then extracted using an in-house method for fat/oil.

The diethyl ether was vortex mixed with NaOH in methanol. Acidified NaBr solution was added and vortex mixed. The top ether layer was evaporated and then hexane added and vortex mixed, the hexane was discarded. This step was repeated. The aqueous layer was extracted 3 times with a mixture of ether and ethyl acetate and these washings combined in a vial containing Na_2SO_4. Phenyl boronic acid was added and this was heated at 60˚C for 30 minutes. After cooling, the sample was evaporated to dryness, then re-dissolved in isooctane before being transferred to a vial for GCMS analysis.

Samples were analysed alongside calibration standards, which were used to quantify any residues detected. Identification of the analytes was confirmed by comparison of retention times and peak area ion ratios with those of the calibration standards using acceptance criteria specified in SANTE/2020/12830 Rev.1 (16) and retained Regulation (EU) 2019/2093 (17). Isotopically labelled internal standards were included in the analysis.

Multiple spiked samples and an in-house reference sample were also included in the analytical batch.

6.7 Isoflavone analyses

A subset of samples was analysed for isoflavones. The samples analysed were the soya drinks and soya-based infant formula. The analysis was carried out for isoflavone glycosides (genistin, daidzin, and glycitin), and the aglycones (genistein, daidzein, and glycitein) and total isoflavones. The method was a modified version of an instrument vendor application note (18).

An aliquot (1 mL) of the liquid sample was diluted to a total volume of 10 mL with a mixture of acetonitrile : water (70:30, v/v) in a centrifuge tube. Samples were shaken, vortex mixed then centrifuged for 5 minutes at 5000 rpm. The supernatant was collected, and an aliquot filtered through a 25 mm nylon syringe filter (0.45 µm) before analysis by HPLC with UV detection at 254 nm.

The following HPLC conditions were used:

Column: YMC-Pack ODS-AM 250mm x 3.0 mm x 5µm

Flow rate: 1 ml/min

Mobile phase: A = 0.05% Trifluoroacetic acid (TFA) aq, B = 0.05% TFA MeOH

The gradient profile used is given in Table 4.

Table 4. Gradient profile for isoflavone analysis:

Time (minutes)	% Mobile phase A	% Mobile phase B
0	90	10
1	90	10
8	65	35
12	65	35
12.1	90	10
18	90	10

7. Results and Discussion

7.1 Mycotoxins analyses – oat products by 11+ method

The results for the oat products are given in Table 6. None of the samples contained mycotoxins above maximum levels that are currently in force from Retained Commission Regulation (EC) No 1881/2006 (1, 2, 3). All other results were very low concentration or below the LOQ for the method.

Aflatoxins, fumonisins, α-ZOL and β-ZOL were not detected in any oat product sample. Two samples contained a low level of sterigmatocystin, one at 0.3 µg/kg and one just below the LOQ of 0.25 µg/kg. Seven samples contained OTA, at levels from 0.3 to 1.8 µg/kg, and two contained samples contained trace levels just below the LOQ (0.25 µg/kg). None of these samples exceeded the maximum level for OTA of 3 µg/kg.

Two samples contained ZON, one at 3.9 µg/kg and the other at 2.8 µg/kg.

The most frequently found mycotoxin was DON, eleven samples contained levels from 6.1 to 233 µg/kg, and another five samples contained very low levels that were below the LOQ. The sample with the highest DON level (S21-040000 – porridge oats), also contained the highest 3-AcDON (37.5 µg/kg), HT-2 (45.2 µg/kg), and T-2 (10.9 µg/kg) levels. It also contained the highest level of T2-3α-G, although this is an indicative value of 4.2 µg/kg as it was just below the LOQ of 5 µg/kg. Two samples contained 3-AcDON, these were also the samples with highest DON and HT-2 concentrations.

Eight samples contained HT-2 above the LOQ (levels from 7.5 to 45.2 µg/kg) and three samples contained T-2 (8.5 to 10.9 µg/kg), in both cases several samples were observed to contain low levels below the LOQ, these are reported for completeness in Table 6, but are not quantitative.

Quality control data for these analyses is given in Table 7. An in-house reference sample analysed in the batch gave values that were very close to the assigned value for the analytes it contained.

7.2 Mycotoxins analyses – non-dairy alternative products by 11+ method validation

The 11+ method had not been used for analysis of non-dairy alternative drinks previously, therefore some method evaluation and in-house validation were carried out. The method described in section 5.5 was assessed for oat, almond, soya and coconut drinks by analysis of replicate samples spiked at two levels, 1x LOQ and 50x LOQ (see Table 2). Four replicates at each level for each product were analysed and the results are given in Table 8 to Table 11.

For the analytes that the IAC was stated to be applicable for satisfactory recovery (60-110%) was obtained in most cases, except for ZON in soya drinks. This recovery range is a generally accepted range for mycotoxin analysis (15), although Regulation (EC) 401/2006 does allow recovery from 60-130% for the analytes included in this method (15). Additional toxins that also achieved satisfactory performance were 3-AcDON, T2α3-G, α-ZOL and β -ZOL. Recovery for DON-3-G was low at around 20%, and sterigmatocystin was also low at 20-37%. Soya products gave consistently lower recovery for ZON, the reason for this is not clear. ZON and isoflavones both exhibit oestrogenic effects due to their structural similarities, and genistein and ZON have been reported to produce synergist oestrogenic effects in in-vitro (19). Therefore, it is possible there may be some element of competition for binding for the ZON antibodies within the IAC taking place for soya samples.

7.3 Mycotoxins analyses – non-dairy alternative drinks by 11+ method

The *Fusarium* mycotoxin results for the non-dairy alternative drinks are given in Table 12 and the results for the other mycotoxins are given in Table 13. Recoveries for STG and DON-3-G were low, however, recoveries for ZON, α-ZOL and β-ZOL were also lower than found during validation. These results are reported not corrected and are for information only. No ZON compounds were found above the LOQ. Three samples contained a very low level of ZON (below LOQ). One sample (S21-040025) a soya drink also contained DON (1.56 µg/kg), aflatoxin B1 (0.005 µg/kg) and fumonisin B1 (0.148 µg/kg).

Five samples contained low levels of AFB1, no other aflatoxins were detected. OTA, STG, and FB3 were not detected in any sample, one sample contained a low level of FB2.

DON was detected in ten samples at levels from 0.15 to 8.78 µg/kg, another six samples contained trace levels below the LOQ. The four samples with the highest DON levels also contained DON-3-G at levels from 0.3 to 0.93 µg/kg (uncorrected) and two of these samples also contained 3-AcDON. These four samples also contained the highest HT-2 and T-2

levels, three contained T-2 above the LOQ. The sample with highest DON, HT-2 and T-2 levels (S21-040027) also contained T-2-3α-G above the LOQ. These four samples were all oat drinks.

7.4 Mycotoxins analyses – oat products by accredited multi mycotoxin method - In-house method FSG 818

In order to obtain results for all analytes requested a second method was used to analyse the samples. The results are given in Table 14. The results for analytes that are measured by both methods are similar. This method has higher LOQs than the 11+ IAC method. Two samples (S21-040004 and S21-04022) were lost due to instrument issues. There was insufficient time to re-analyse these samples by this method. Therefore, there are no results for these samples by this method, meaning there is no nivalenol result for these samples, or a quantitative result for DON-3-G.

NIV was not detected in any sample, although the LOQ is quite high at 50 µg/kg, it is an extremely difficult compound to analyse and ionises poorly giving a much lower response than the other trichothecenes.

Results for DON, 3-AcDON, ZON, HT-2 and T-2 were comparable and followed the same pattern as the results from the 11+ method. Similarly results for T2-3α-G were also similar although they were below the LOQ for this method.

DON-3-G was found in five samples, the highest level was found in sample S21-040000 that contained the highest DON level. In addition, another three samples contained low levels below the LOQ (Table 14). One sample was found to contain 15-AcDON at 54 µg/kg although no other mycotoxins were found in this sample.

Other trichothecenes measured using this method: NEO, FUSX, and DAS and glucosides of other *Fusarium* mycotoxins ZON, α-ZOL and β-ZOL were not detected.

7.5 Mycotoxins analyses – non-dairy alternative drinks by accredited multi mycotoxin method - In-house method FSG 818

Non-dairy alternative drinks were also analysed by the second LC-MS/MS method. The LOQs for this method are much higher than the 11+ method and no residues were detected

for any analytes except sample S21-040027 where residues of DON and DON-3-G were observed. Over spikes of all analytes were measured, although recovery was lower than expected for some analytes, e.g. DAS, FUSX and T-2-3α-G, recovery for DON, NIV, 3-AcDON, 15-AcDON, and HT-2 were all within the expected range and quantitative results had been obtained for T-2-3α-G by the 11+ method.

7.6 Comparison of T-2 and HT-2 results to proposed maximum levels

There are currently no maximum levels in force for T-2 and HT-2, however there is a proposal within the European Union to introduce a maximum level of 20 µg/kg for the sum of T-2 and HT-2 in 'pastries, biscuits, cereal snacks, breakfast cereals including formed cereal flakes' and for an ML of 50 µg/kg for 'cereal grains placed on the market for the final consumer, including oats' (6, 7).

Sample S21-040000, a porridge oats sample, contained 56.1 µg/kg sum T-2 and HT-2 by the 11+ method and 73.2 µg/kg by method FSG 818. Taking Measurement Uncertainty (MU) into account, this result is not an exceedance of the proposed maximum level of 50 µg/kg by the 11+ method (56.1 ± 16.8 µg/kg) but could be an exceedance from the result of the method FSG 818 (73.2 ± 16.1 µg/kg). Further repeat analysis would be required to obtain a mean result by one method (usually n=3) to confirm this.

Samples S21-040014 and S21-040009 were both porridge oats samples. These samples contained 23 µg/kg and 29 µg/kg by the 11+ method, and 24.6 µg/kg and 33 µg/kg by method FSG 818, therefore both would be below the proposed ML.
Sample S21-040022 was a sample of oat cakes, it was found to contain 23.9 µg/kg by the 11+ method, with MU taken into account it was not an exceedance (23.9 ± 7.17 µg/kg) of the proposed ML of 20 µg/kg for biscuits and snacks. These results are summarised in Table 15.

7.7 3-MCPD and Glycidyl esters analyses of coconut drinks

Five samples of coconut drinks were analysed for 3-MCPD esters and 2-MCPD esters. During analysis these are converted to the free form 2 and 3-MCPD and detected as such. Glycidyl esters are converted to 3-MBPD (monobromopropanediol). For glycidols, 10 µg/kg free form glycidol will be equivalent to 10 µg/kg detected 3-MBPD.

The results are given in Table 16 and corresponding quality control results in Table 17. The analysis gave satisfactory performance for an in-house reference material, the repeatability for 2-MCPD was quite high, but was just at the limit of acceptable measurement uncertainty (of 44%) for this analysis (17). No 3-MCPD, 2-MCPD or glycidyl esters were detected in the samples with a LOQ of 5 µg/kg for each compound. The results for 3-MCPD are accredited, but 2-MCPD and glycidyl esters are not.

7.8 Isoflavone analysis of soya products

Eight samples of soya drink and two samples of soya based infant formula were analysed for isoflavone glycosides (genistin, daidzin, and glycitin), aglycones (genistein, daidzein, and glycitein) and total isoflavones. The results are given in Table 18 and are not accredited. The method was a simple method, that used the 'dilute and shoot' principle, samples were simply diluted, centrifuged and filtered before injection on the HPLC system (18).

Genistin was found at the highest level in all samples at levels from 133 to 304 mg/L. Daidzin was the next most abundant compound at levels from 41 to 121 mg/L, again it was present in all samples. Genistein was also measured in all samples (1.3 to 18.2 mg/L), daidzein was found in all but one sample at levels from 1.3 to 13.5 mg/L. Glycitin was detected in five samples and glycitein was only detected in one sample of infant formula. Total isoflavone levels ranged from 213 to 488 mg/L (mg/kg for infant formula). These compounds are natural constituents of soya products, there are no maximum levels or guidance values. It is difficult to find good data on isoflavone levels in soya milk, however one recent publication reported mean genistein content of 17.58 ± 8.38 µg/mL (equivalent to 17.58 mg/L), which is comparable with these results (19). Another publication reported levels of genistein of 25.86 mg/L and daidzein 8.25 mg/L (20), again comparable with the results of this study

7.9 Additional analysis of non-dairy alternative drinks samples

FSS had undertaken another survey of oat products, samples had been submitted to Official Laboratories. At the request of FSS a small subset of these samples, all non-dairy alternative drinks made from oats, were analysed for mycotoxins including modified forms. In total 17 mycotoxins including DON, NIV, T-2, HT-2 and ZON, as well as modified forms of DON, T-2 and ZON compounds were analysed. No residues of any mycotoxin were found above the LOQ, and in fact no residues were detected between the LOQ and LOD as had been observed for some of the other samples. The quality control results for this analysis were

good, average recoveries (n=6) ranged from 69-100%, except for 15-Ac-DON where average recovery was 150% and for ZON, α-ZOL and β-ZOL where it averaged ca 40%. The results for these samples are given in Annex D., Table 19.

8. Summary and Conclusions

8.1 Two methods were used to analyse the samples to give results for all the requested mycotoxin analytes. The use of two methods allowed additional mycotoxins to be analysed in the survey samples as well as the mycotoxins requested by FSS. Mycotoxins were detected in several oat products, although no sample exceeded any maximum level from Retained Commission Regulation (EC) No 1881/2006 (1, 2, 3).

The most frequently found mycotoxin was DON, eleven samples contained levels from 6.1 to 233 µg/kg. The sample with the highest DON level (S21-040000 – porridge oats), also contained the highest 3-AcDON (37.5 µg/kg), HT-2 (45.2 µg/kg), and T-2 (10.9 µg/kg) levels. It also contained a low level of T-2-3α-G. Eight samples contained HT-2 above the LOQ (levels from 7.5 to 45.2 µg/kg) and three samples contained T-2 (8.5 to 10.9 µg/kg).

DON-3-G was found in five samples, the highest level was found in the sample that contained the highest DON level. Two samples contained 3-AcDON, these were also the samples with highest DON and HT-2 concentrations.

The ratio of T-2-3α-G to T-2 ranged from 30-39%, except for one sample with very low levels of both analytes (S21-040014, both below LOQ) where the ratio was 64 %, a higher level of variability would be expected at this level.

The ratio of DON-3-G to DON ranged from 30-76% where residues were detected, except for sample S21-039998 where the DON-3-G level was 129% of the DON level. The levels found for this sample were very low, both DON-3-G and DON were less than 40 µg/kg.

No modified forms of ZON were detected, it was not possible to analyse for modified HT-2 due to the lack of a commercially available analytical standard.

8.2 Mycotoxins were also detected in the non-dairy alternative drinks. Four oat drinks contained the highest levels of DON and also contained DON-3-G at levels from 0.3 to 0.93 µg/kg (uncorrected). These four samples also contained the highest HT-2 and T-2 levels. The sample with the highest DON, HT-2 and T-2 levels also contained T-2-3α-G above the LOQ. It was more difficult to calculate ratios of parent to modified mycotoxins for

these samples, for the two samples with measurable T-2-3α-G and T-2 the ratios were 30% and 90%. For DON and DON-3-G, results from the 11+ method, the ratios were around 10-20%, although this will be an underestimate as the results for DON-3-G were not corrected for recovery. For one sample found to contain both analytes using the other method that had a higher LOQ, the ratio was 105%. Again, this must be viewed with caution as both results were below the LOQ for this method so should be viewed as indicative. Low levels of AFB1 were detected in some samples, one soya drink also contained DON, and fumonisin B1 as well as a trace level of ZON.

8.3 There were frequent observations of analytes just below the current LOQ in all types of non-dairy alternative drinks samples. If required this LOQ could be reduced further by carrying out a sample concentration step after IAC clean-up, although that may cause a decrease in analytical recovery. More work may be required to improve the current multi-mycotoxin analytical method to obtain lower LOQs for some compounds e.g. NIV for the non-dairy alternative drinks samples.

8.4 No 3-MCPD or glycidyl esters were detected in the five coconut samples analysed.

8.5 Isoflavones were measured in soya products using a simple method, with HPLC-UV analysis. Total isoflavone content ranged from 213 to 488 mg/L (mg/kg for infant formula). There are no MLs for these natural constituents of soya, but the levels found are in agreement with other published findings.

8.6 This study has shown the potential for the occurrence of multi-mycotoxins in Scottish oat products and non-dairy alternative drinks. Modified forms as glucosides of T-2 and DON were detected, as well as 3-AcDON and 15-AcDON. No samples in this survey contained any mycotoxin above a current in force maximum level. In most cases the levels of modified forms were very low and below the LOQ of the methods used. Where they were quantified they were present at varying proportions of the parent toxin, averaging at approximately 35% for the ratio of T-2-3α-G to T-2 and from 30-76% for DON-3-G to DON. There were two potential exceedances of proposed MLs for the sum of T-2 and HT-2 toxins. Sample S21-040000, a porridge oats sample, contained 56.1 µg/kg sum T-2 and HT-2 by the 11+ method and 73.2 µg/kg by method FSG 818, and sample S21-040022, a sample of oat cakes, which was found to contain 23.9 µg/kg. When measurement uncertainty was taken into account

S21-040000 would not be considered an exceedance of the proposed ML of 50 µg/kg for cereals sold direct to the consumer for the result by the 11+ method, but it would by method FSG 818. Sample S21-040022 would not be considered to exceed the proposed ML of 20 µg/kg for biscuits and snacks.

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Annex A: Summarised sample information

Table 5. Summarised sample information

Fera Sample No.	Product Category	General Product Description
S21-040000	Porridge/Oatmeal/Bran	Scottish porridge oats
S21-039999	Porridge/Oatmeal/Bran	Scottish oatmeal
S21-039998	Porridge/Oatmeal/Bran	Scottish Porridge Oat + Bran
S21-040033	Porridge/Oatmeal/Bran	Scottish Oats
S21-040004	Porridge/Oatmeal/Bran	Whole Scottish porridge oats
S21-040014	Porridge/Oatmeal/Bran	Porridge oats
S21-040009	Porridge/Oatmeal/Bran	Fine Scotch Oatmeal
S21-040007	Granola and Muesli	Granola
S21-040024	Granola and Muesli	Scottish oats organic granola
S21-039992	Granola and Muesli	Scottish oat muesli
S21-040026	Oatcakes	Oatcakes
S21-040015	Oatcakes	Fine milled oatcakes
S21-039994	Oatcakes	Rough cut oatcakes
S21-040022	Oatcakes	Scottish Rough Oatcakes
S21-040005	Oat biscuits	Oat Biscuits (sweet)
S21-040006	Oat biscuits	Oat Biscuits (sweet)
S21-040010	Oat biscuits	Oat Biscuits (sweet)
S21-040027	Oat drinks	Scottish oat drink
S21-040019	Oat drinks	UHT oat drink
S21-040008	Oat drinks	Oat drink
S21-040017	Oat drinks	Oat drink unsweetened

Table 5. contd. Summarised sample information

Fera Sample No.	Product Category	General Product Description
S21-040021	Soya drinks (unsweetened)	UHT Soya drink
S21-039997	Soya drinks (unsweetened)	Soya drink unsweetened
S21-040029	Soya drinks (unsweetened)	Unsweetened soya milk alternative
S21-040013	Soya drinks (unsweetened)	Longlife Unsweetened Soya Milk Alternative
S21-040030	Soya drinks (unsweetened)	Soya drink for children from 1 year, with added minerals and vitamins.
S21-040003	Soya drinks (unsweetened)	Unsweetened soya drink
S21-040012	Soya drinks (unsweetened)	Unsweetened soya drink
S21-040025	Soya drinks (unsweetened)	Soya drink
S21-039993	Soya formula	Soya infant formula
S21-040023	Soya formula	Soy based infant formula
S21-040016	Coconut drinks	Coconut drink
S21-040011	Coconut drinks	Coconut drink UHT
S21-039995	Coconut drinks	Sweetened coconut drink
S21-040032	Coconut drinks	Longlife coconut drink
S21-040001	Coconut drinks	Coconut drink
S21-040020	Almond drinks	Unsweetened almond drink
S21-039996	Almond drinks	Dairy Free almond drink
S21-040028	Almond drinks	Unsweetened almond milk alternative
S21-040031	Almond drinks	Almond no sugars almond drink
S21-040002	Almond drinks	Unsweetened almond drink

Table 6. Mycotoxin results for oat products using 11+ method, corrected for recovery

	3-AcDON	DON	T2	HT2	T2-a3-Gl	ZON	a-ZOL	b-ZOL
Sample name	**Concentration, corrected for recovery, µg/kg**							
S21-039992	<5	6.1	<5	<5 (3.5q)	<5	<2.5	<2.5	<2.5
S21-039994	<5 (1.6q)	12.4	<5 (1.5q)	<5 (3.3q)	<5	<2.5	<2.5	<2.5
S21-039998	<5 (2.0q)	19.7	<5 (1.9q)	7.5	<5	<2.5	<2.5	<2.5
S21-039999	<5 (2.4q)	27.6	<5 (3.9q)	13.6	<5 (1.2q)	<2.5	<2.5	<2.5
S21-040000	37.5	233	10.9	45.2	<5 (4.2q)	2.8	<2.5	<2.5
S21-040004	<5	8.3	<5 (1.7q)	10.8	<5 (1.2q)	<2.5	<2.5	<2.5
S21-040005	<5	<5 (2.4q)	<5	<5 (3.0q)	<5	3.9	<2.5	<2.5
S21-040006	<5	<5 (2.5q)	<5	<5 (2.6q)	<5	<2.5	<2.5	<2.5
S21-040007	<5	<5 (3.6q)	<5	<5 (3.1q)	<5	<2.5	<2.5	<2.5
S21-040009	<5	24.8	10.0	19.0	<5 (3.1q)	<2.5	<2.5	<2.5
S21-040010	<5	<5 (3.6q)	<5	<5 (1.7q)	<5	<2.5	<2.5	<2.5
S21-040014	12.2	63.6	<5 (2.4q)	21.0	<5 (1.6q)	<2.5 (0.7q)	<2.5	<2.5
S21-040015	<5	6.5	<5	<5 (2.2q)	<5	<2.5	<2.5	<2.5
S21-040022	<5	<5	8.5	15.4	<5	<2.5	<2.5	<2.5
S21-040024	<5	<5 (3.3q)	<5	<5	<5	<2.5	<2.5	<2.5
S21-040026	<5	8.9	<5	<5 (3.6q)	<5	<2.5	<2.5	<2.5
S21-040033	<5 (1.6q)	12.9	<5 (2.1q)	13.4	<5 (1.7q)	<2.5	<2.5	<2.5

q – results are non-quantitative and for information only as below the LOQ.

Table 6. Contd. Mycotoxin results for oat products using 11+ method, corrected for recovery

	AFB1	AFB2	AFG1	AFG2	OTA	STG	FB1	FB2	FB3
Sample name	**Concentration, corrected for recovery, µg/kg**								
S21-039992	<0.25	<0.25	<0.25	<0.25	<0.25	<0.25	<10	<5	<5
S21-039994	<0.25	<0.25	<0.25	<0.25	0.5	<0.25	<10	<5	<5
S21-039998	<0.25	<0.25	<0.25	<0.25	<0.25	<0.25	<10	<5	<5
S21-039999	<0.25	<0.25	<0.25	<0.25	<0.25	<0.25	<10	<5	<5
S21-040000	<0.25	<0.25	<0.25	<0.25	<0.25	<0.25	<10	<5	<5
S21-040004	<0.25	<0.25	<0.25	<0.25	<0.25	<0.25	<10	<5	<5
S21-040005	<0.25	<0.25	<0.25	<0.25	<0.25	<0.25	<10	<5	<5
S21-040006	<0.25	<0.25	<0.25	<0.25	<0.25	<0.25	<10	<5	<5
S21-040007	<0.25	<0.25	<0.25	<0.25	<0.25	<0.25 (0.2g)	<10	<5	<5
S21-040009	<0.25	<0.25	<0.25	<0.25	0.6	<0.25	<10	<5	<5
S21-040010	<0.25	<0.25	<0.25	<0.25	0.8	<0.25	<10	<5	<5
S21-040014	<0.25	<0.25	<0.25	<0.25	0.7	<0.25	<10	<5	<5
S21-040015	<0.25	<0.25	<0.25	<0.25	<0.25 (0.2g)	<0.25	<10	<5	<5
S21-040022	<0.25	<0.25	<0.25	<0.25	1.7	0.3	<10	<5	<5
S21-040024	<0.25	<0.25	<0.25	<0.25	1.8	<0.25	<10	<5	<5
S21-040026	<0.25	<0.25	<0.25	<0.25	<0.25 (0.2g)	<0.25	<10	<5	<5
S21-040033	<0.25	<0.25	<0.25	<0.25	0.3	<0.25	<10	<5	<5

Table 7. Quality Control results for analysis of oat products by 11+ method

	3-AcDON	DON	T2	HT2	T2-a3-Gl	ZON	a-ZOL	b-ZOL
	Concentration, corrected for recovery, µg/kg							
IHR 04409 result	<5	<5	192.8	260.8	<5	<2.5	<2.5	<2.5
IHR 04409 assigned values			195	291				
low limit (Z-score -2)			108.2	163.6				
high limit (Z-score +2)			281.8	418.4				
Average recovery, %, (n=5)	90	76	92	90	90	79	66	91

	AFB1	AFB2	AFG1	AFG2	OTA	STG	FB1	FB2	FB3
	Concentration, corrected for recovery, µg/kg								
IHR 04409 result	3.4	2.8	1.8	1.0	4.3	9.8	<10	<5	<5
IHR 04409 assigned values	3.44	2.52	1.86	0.98	3.57	9.6			
low limit (Z-score -2)	1.85	1.35	0.97	0.54	1.86	5.1			
high limit (Z-score +2)	5.03	3.69	2.75	1.43	5.28	14.1			
Average recovery, %, (n=5)	88	88	71	84	77	42	101	111	143

Table 8.Validation data for oat milk – 11+ IAC method

	AFB1	AFB2	AFG1	AFG2	FB1	FB2	FB3	OTA	STG
Sample name	**Recovery (%)**								
spike level ug/kg	0.01	0.01	0.01	0.01	0.4	0.2	0.2	0.01	0.01
Oat Sp1 low	65.1	76.3	79.9	76.7	78.5	91.8	85.7	113.9	20.2
Oat Sp2 low	85.2	94.1	100.4	88.5	99.9	99.8	97.2	109.4	21.7
Oat Sp3 low	70.0	81.0	74.2	82.0	82.8	89.4	79.0	83.0	17.4
Oat Sp4 low	71.3	79.2	81.4	82.8	66.3	59.3	68.1	100.4	18.2
average (n=4)	72.9	82.7	84.0	82.5	81.9	85.1	82.5	101.7	19.4
S.d.	8.6	7.9	11.4	4.8	13.9	17.7	12.2	13.7	1.9
cv	11.8	9.5	13.5	5.9	17.0	20.8	14.8	13.4	9.9
spike level ug/kg	0.5	0.5	0.5	0.5	20.0	10.0	10.0	0.5	0.5
Oat Sp1 High	85.0	88.9	80.5	99.1	95.0	107.6	134.8	68.6	11.8
Oat Sp2 High	94.9	100.0	87.9	107.4	105.3	120.7	151.9	77.8	13.3
Oat Sp3 High	87.8	92.1	80.4	102.1	100.4	113.0	142.3	72.8	12.6
Oat Sp4 High	74.9	82.1	34.6	63.4	95.4	103.5	135.0	65.4	10.0
average (n=4)	85.6	90.8	70.8	93.0	99.0	111.2	141.0	71.2	11.9
S.d.	8.3	7.4	24.4	20.0	4.9	7.4	8.1	5.4	1.4
cv	9.7	8.2	34.5	21.5	4.9	6.7	5.7	7.5	12.0

Highlighted cells for STG as recovery outside acceptable range.

Table 8. Contd. Validation data for oat milk – 11+ IAC method

	DON	3-AcDON	DON-3-G	T2	HT2	T2-α3-G	ZON	α-ZOL	β-ZOL
Sample name	**Recovery (%)**								
spike level ug/kg	0.2	0.2	0.1	0.2	0.2	0.2	0.1	0.1	0.1
Oat Sp1 low	105.0	102.4	20.9	99.9	83.6	97.6	60.8	81.5	74.8
Oat Sp2 low	150.9	119.8	33.0	126.6	112.2	113.0	71.9	96.4	99.9
Oat Sp3 low	94.5	97.9	12.9	102.7	82.4	92.9	59.2	78.7	88.5
Oat Sp4 low	120.6	112.6	15.3	99.7	86.7	105.3	60.3	85.5	97.5
average (n=4)	117.8	108.2	20.5	107.2	91.2	102.2	63.1	85.5	90.2
S.d.	24.5	9.9	9.0	13.0	14.1	8.8	5.9	7.8	11.4
cv	20.8	9.1	43.7	12.1	15.5	8.6	9.4	9.1	12.6
spike level ug/kg	10.0	10.0	5.0	10.0	10.0	10.0	5.0	5.0	5.0
Oat Sp1 High	79.0	119.2	7.1	88.2	85.4	94.6	72.3	74.3	85.9
Oat Sp2 High	92.4	126.2	10.2	101.7	101.3	106.8	85.2	91.7	102.9
Oat Sp3 High	86.1	118.1	10.4	94.6	90.0	101.8	78.5	81.4	96.1
Oat Sp4 High	89.9	116.0	8.9	90.2	87.5	97.2	64.8	71.7	87.3
average (n=4)	86.9	119.9	9.2	93.7	91.1	100.1	75.2	79.8	93.1
S.d.	5.8	4.4	1.5	6.0	7.1	5.3	8.7	9.0	8.0
cv	6.7	3.7	16.5	6.4	7.8	5.3	11.6	11.2	8.6

Highlighted cells for DON-3-G as recovery outside acceptable range.

Table 9. Validation data for almond milk – 11+ IAC method

	AFB1	AFB2	AFG1	AFG2	FB1	FB2	FB3	OTA	STG
Sample name	**Recovery (%)**								
spike level ug/kg	0.01	0.01	0.01	0.01	0.4	0.2	0.2	0.01	0.01
Almond Sp 1 low	95.7	93.0	95.8	77.2	79.5	89.7	82.0	91.4	41.9
Almond Sp 2 low	90.0	88.1	95.3	77.6	71.6	76.4	75.8	80.1	32.6
Almond Sp 3 low	94.8	95.5	100.2	78.7	77.7	80.0	81.5	97.2	38.8
Almond Sp 4 low	90.1	90.1	100.3	76.2	45.1	54.1	49.1	111.6	36.7
average (n=4)	92.6	91.7	97.9	77.4	68.5	75.0	72.1	95.1	37.5
S.d.	3.0	3.3	2.7	1.0	16.0	15.1	15.6	13.1	3.9
cv	3.2	3.5	2.8	1.3	23.3	20.1	21.6	13.8	10.5
spike level ug/kg	0.5	0.5	0.5	0.5	20.0	10.0	10.0	0.5	0.5
Almond Sp 1 high	84.0	86.5	77.2	90.6	88.2	96.6	118.6	72.1	27.4
Almond Sp 2 high	86.4	90.3	79.1	90.6	94.7	104.2	129.0	72.8	27.0
Almond Sp 3 high	87.8	92.4	81.8	94.7	93.7	102.5	127.6	74.0	28.4
Almond Sp 4 high	83.5	87.0	60.4	76.0	76.2	79.2	100.2	67.6	21.5
average (n=4)	85.4	89.1	74.6	88.0	88.2	95.6	118.8	71.6	26.1
S.d.	2.0	2.8	9.7	8.2	8.5	11.4	13.3	2.8	3.1
cv	2.4	3.2	13.0	9.3	9.6	12.0	11.2	3.9	11.9

Highlighted cells for STG as recovery outside acceptable range.

Table 9. Contd. Validation data for almond milk – 11+ IAC method

	DON	3-AcDON	DON-3-G	T2	HT2	T2-α3-G	ZON	α-ZOL	β-ZOL
Sample name	**Recovery (%)**								
spike level ug/kg	0.2	0.2	0.1	0.2	0.2	0.2	0.1	0.1	0.1
Almond Sp 1 low	64.5	105.4	13.4	63.0	94.9	93.3	96.7	97.7	102.8
Almond Sp 2 low	57.0	95.1	22.8	60.5	90.1	85.8	78.8	102.0	99.5
Almond Sp 3 low	60.6	120.1	28.7	66.4	91.0	87.7	88.0	93.1	104.4
Almond Sp 4 low	64.8	96.9	22.7	64.7	105.0	80.6	85.2	95.2	115.6
average (n=4)	61.7	104.4	21.9	63.6	95.3	86.9	87.2	97.0	105.6
S.d.	3.7	11.4	6.3	2.5	6.8	5.2	7.4	3.8	7.0
cv	6.0	10.9	28.9	3.9	7.2	6.0	8.5	3.9	6.6
spike level ug/kg	10.0	10.0	5.0	10.0	10.0	10.0	5.0	5.0	5.0
Almond Sp 1 high	77.2	107.4	12.5	84.6	82.6	90.2	87.7	85.6	94.9
Almond Sp 2 high	78.1	108.7	11.2	87.5	87.0	92.5	93.2	88.3	100.1
Almond Sp 3 high	78.6	112.2	11.2	88.4	86.3	98.6	91.2	88.2	101.0
Almond Sp 4 high	85.8	113.7	7.8	87.4	90.6	90.4	89.9	89.9	98.5
average (n=4)	79.9	110.5	10.7	86.9	86.6	92.9	90.5	88.0	98.6
S.d.	4.0	2.9	2.0	1.6	3.3	3.9	2.3	1.8	2.7
cv	5.0	2.6	18.6	1.9	3.8	4.2	2.6	2.0	2.7

Highlighted cells for DON-3-G as recovery outside acceptable range.

Table 10. Validation data for soya milk – 11+ IAC method

	AFB1	AFB2	AFG1	AFG2	FB1	FB2	FB3	OTA	STG
Sample name	**Recovery (%)**								
spike level ug/kg	0.01	0.01	0.01	0.01	0.4	0.2	0.2	0.01	0.01
Soya Sp1 low	-	67.9	68.5	72.4	76.0	72.1	82.8	99.5	25.9
Soya Sp2 low	-	87.5	84.1	91.1	91.9	89.9	91.8	136.0	20.3
Soya Sp3 low	-	66.4	65.4	64.8	73.8	67.9	76.2	102.4	21.8
Soya Sp4 low	-	65.9	64.8	64.7	60.9	65.0	60.8	88.7	17.5
average (n=4)	-	71.9	70.7	73.3	75.6	73.7	77.9	106.7	21.4
S.d.	-	10.4	9.1	12.4	12.7	11.2	13.1	20.4	3.5
cv	-	14.5	12.8	17.0	16.8	15.2	16.8	19.1	16.5
spike level ug/kg	0.5	0.5	0.5	0.5	20.0	10.0	10.0	0.5	0.5
Soya Sp1 high	74.6	79.3	71.4	82.6	87.3	93.4	117.1	62.2	14.4
Soya Sp2 high	75.4	81.1	73.9	83.9	93.1	97.7	123.0	61.1	14.3
Soya Sp3 high	100.4	111.5	96.6	114.9	128.4	134.8	173.1	87.7	13.5
Soya Sp4 high	72.1	79.8	55.7	72.4	91.6	95.0	122.8	64.4	12.2
average (n=4)	80.6	87.9	74.4	88.4	100.1	105.2	134.0	68.9	13.6
S.d.	13.3	15.8	16.8	18.4	19.0	19.8	26.2	12.7	1.0
cv	16.5	17.9	22.6	20.8	19.0	18.8	19.5	18.4	7.7

- Not possible to calculate recovery due to residue in blank

Highlighted cells for STG as recovery outside acceptable range.

Table 10. Contd. Validation data for soya milk – 11+ IAC method

	DON	3-AcDON	DON-3-G	T2	HT2	T2-α3-G	ZON	α-ZOL	β-ZOL
Sample name	**Recovery (%)**								
spike level ug/kg	0.2	0.2	0.1	0.2	0.2	0.2	0.1	0.1	0.1
Soya Sp1 low	79.9	95.5	22.2	55.1	114.3	94.7	39.2	55.7	72.7
Soya Sp2 low	108.1	105.8	19.2	72.1	130.1	117.3	35.7	59.5	84.0
Soya Sp3 low	83.8	86.7	13.6	50.7	90.7	85.3	38.0	60.1	75.0
Soya Sp4 low	75.1	88.0	11.7	49.8	98.3	79.6	19.8	40.1	53.9
average (n=4)	86.7	94.0	16.7	56.9	108.3	94.2	33.2	53.8	71.4
S.d.	14.7	8.8	4.9	10.4	17.5	16.6	9.0	9.4	12.6
cv	17.0	9.3	29.3	18.3	16.2	17.6	27.2	17.4	17.7
spike level ug/kg	10.0	10.0	5.0	10.0	10.0	10.0	5.0	5.0	5.0
Soya Sp1 high	74.2	104.3	5.5	73.7	84.8	85.6	40.6	45.5	62.7
Soya Sp2 high	77.4	109.3	6.2	76.6	83.7	94.2	46.1	49.2	69.3
Soya Sp3 high	100.9	145.2	10.4	103.4	117.3	128.8	53.6	60.3	98.0
Soya Sp4 high	84.8	109.6	5.8	77.1	87.1	97.8	34.1	39.1	63.9
average (n=4)	84.3	117.1	7.0	82.7	93.2	101.6	43.6	48.5	73.5
S.d.	11.9	18.9	2.3	13.9	16.1	18.9	8.3	8.9	16.6
cv	14.1	16.1	33.3	16.8	17.3	18.6	19.0	18.3	22.6

Highlighted cells for DON-3-G as recovery outside acceptable range.

Table 11. Validation data for coconut drink – 11+ IAC method

	AFB1	AFB2	AFG1	AFG2	FB1	FB2	FB3	OTA	STG
Sample name	**Recovery (%)**								
spike level ug/kg	0.01	0.01	0.01	0.01	0.4	0.2	0.2	0.01	0.01
Coconut Sp1 low	71.8	77.0	75.0	75.6	70.4	68.5	73.2	53.2	35.5
Coconut Sp2 low	63.8	73.1	72.2	64.9	63.7	66.7	69.5	50.4	32.3
Coconut Sp3 low	65.4	72.5	71.3	62.8	68.3	62.6	69.4	66.4	29.2
Coconut Sp4 low	65.3	72.6	75.1	68.5	42.8	38.0	36.8	55.0	29.2
average (n=4)	66.6	73.8	73.4	68.0	61.3	58.9	62.2	56.3	31.5
S.d.	3.5	2.2	2.0	5.6	12.6	14.2	17.0	7.0	3.0
cv	5.3	2.9	2.7	8.3	20.6	24.0	27.4	12.5	9.5
spike level ug/kg	0.5	0.5	0.5	0.5	20.0	10.0	10.0	0.5	0.5
Coconut Sp1 high	83.7	89.6	81.5	95.0	92.6	98.4	122.9	68.7	36.2
Coconut Sp2 high	81.0	87.3	81.0	93.8	89.5	97.0	121.1	67.2	32.8
Coconut Sp3 high	86.1	90.6	83.3	93.9	91.6	99.0	123.2	68.1	32.2
Coconut Sp4 high	79.8	84.6	73.5	86.5	83.2	87.0	114.5	63.3	24.9
average (n=4)	82.7	88.0	79.8	92.3	89.2	95.4	120.4	66.8	31.5
S.d.	2.8	2.7	4.3	3.9	4.2	5.6	4.0	2.4	4.8
cv	3.4	3.0	5.4	4.2	4.7	5.9	3.3	3.6	15.1

Highlighted cells for STG as recovery outside acceptable range.

Table 11. Contd. Validation data for coconut drink – 11+ IAC method

	DON	3-AcDON	DON-3-G	T2	HT2	T2-α3-G	ZON	α-ZOL	β-ZOL
Sample name	**Recovery (%)**								
spike level ug/kg	0.2	0.2	0.1	0.2	0.2	0.2	0.1	0.1	0.1
Coconut Sp1 low	52.5	107.3	0.0	62.9	91.5	80.3	73.3	66.0	100.6
Coconut Sp2 low	53.4	96.2	17.9	58.9	67.4	73.3	72.2	70.1	80.6
Coconut Sp3 low	60.4	95.0	11.3	55.0	93.7	72.8	61.0	65.8	83.1
Coconut Sp4 low	55.5	85.5	27.4	54.7	81.9	70.1	58.9	68.4	81.5
average (n=4)	55.4	96.0	14.2	57.9	83.6	74.1	66.4	67.6	86.4
S.d.	3.5	8.9	11.5	3.9	11.9	4.3	7.5	2.1	9.5
cv	6.4	9.3	81.4	6.7	14.3	5.8	11.3	3.1	11.0
spike level ug/kg	10.0	10.0	5.0	10.0	10.0	10.0	5.0	5.0	5.0
Coconut Sp1 high	79.4	111.7	7.1	85.5	89.4	98.0	86.1	83.5	89.8
Coconut Sp2 high	73.3	111.5	6.8	86.1	86.6	94.3	79.6	80.0	86.3
Coconut Sp3 high	80.6	111.5	6.3	87.7	92.4	95.6	83.4	85.8	91.5
Coconut Sp4 high	81.2	105.7	10.7	84.8	86.5	93.3	72.2	79.3	84.8
average (n=4)	78.6	110.1	7.7	86.0	88.7	95.3	80.3	82.2	88.1
S.d.	3.6	3.0	2.0	1.2	2.8	2.0	6.1	3.1	3.1
cv	4.6	2.7	26.3	1.4	3.2	2.1	7.5	3.7	3.5

Highlighted cells for DON-3-G as recovery outside acceptable range.

Table 12. *Fusarium* Mycotoxin results non-dairy alternative products using 11+ method

	DON	3-AcDON	DON-3-G	T2	HT2	T2-α3-G	ZON	α-ZOL	β-ZOL
Sample number	Concentration, corrected for recovery, µg/kg								
S21-039993*	<0.1 (0.06q)	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
S21-039995	0.19	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
S21-039996	<0.1 (0.06q)	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
S21-039997	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
S21-040001	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
S21-040002	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
S21-040003	0.17	<0.1	<0.1	<0.1	<0.1 (0.08q)	<0.1	<0.1 (0.06q)	<0.1	<0.1
S21-040011	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
S21-040012	<0.1	<0.1	<0.1	<0.1	<0.1 (0.09q)	<0.1	<0.1 (0.06q)	<0.1	<0.1
S21-040013	0.13	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
S21-040016	0.15	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
S21-040017	2.57	<0.1	0.34	0.210	0.519	<0.1 (0.07q)	<0.1	<0.1	<0.1

*soya infant formula analysed as liquid prepared 25g in 250ml = 0.1g/ml powder
Highlighted cells for DON3G, ZON, α-ZOL and β-ZOL not corrected for recovery, values with q reported for information as below LOQ.

Table 12. Contd. *Fusarium* Mycotoxin results non-dairy alternative products using 11+ method

	DON	3-AcDON	DON-3-G	T2	HT2	T2-α3-G	ZON	α-ZOL	β-ZOL
Sample number	Concentration, corrected for recovery, µg/kg								
S21-040019	3.07	0.28	0.49	0.135	0.418	<0.1	<0.1	<0.1	<0.1
S21-040020	<0.1 (0.05q)	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
S21-040021	<.1 (0.07q)	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
S21-040023*	<0.1 (0.05q)	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
S21-040025	1.56	<0.1	<0.1 (0.09q)	<0.1	<0.1	<0.1	<0.1 (0.05q)	<0.1	<0.1
S21-040027	8.78	0.99	0.93	0.187	1.784	0.169	<0.1	<0.1	<0.1
S21-040028	<0.1 (0.07q)	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
S21-040029	0.15	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
S21-040030	<0.1	<0.1	<0.1	<0.1	0.000	<0.1	<0.1	<0.1	<0.1
S21-040031	<0.1	<0.1	<0.1	<0.1	0.000	<0.1	<0.1	<0.1	<0.1
S21-040032	<0.1	<0.1	<0.1	<0.1	0.000	<0.1	<0.1	<0.1	<0.1
S21-040008	1.59	<0.1 (0.05q)	0.30	<0.1 (0.09q)	0.420	<0.1	<0.1	<0.1	<0.1
Average recovery, %, (n=4)	78	83	17	72	104	73	51	54	59

*soya infant formula analysed as liquid prepared 25g in 250ml = 0.1g/ml powder

Highlighted cells for DON3G, ZON, α-ZOL and β-ZOL not corrected for recovery, values with q reported for information as below LOQ.

Table 13. Mycotoxin results non-dairy alternative products using 11+ method

	AFB1	AFB2	AFG1	AFG2	FB1	FB2	FB3	OTA	STG**
Sample number	Concentration, corrected for recovery, µg/kg								
S21-039993*	<0.0025	<0.0025	<0.0025	<0.0025	<0.1	<0.05	<0.05	<0.01	<0.01
S21-039995	0.006	<0.0025	<0.0025	<0.0025	<0.1	<0.05	<0.05	<0.01	<0.01
S21-039996	<0.0025	<0.0025	<0.0025	<0.0025	<0.1	<0.05	<0.05	<0.01	<0.01
S21-039997	<0.0025	<0.0025	<0.0025	<0.0025	<0.1	<0.05	<0.05	<0.01	<0.01
S21-040001	<0.0025	<0.0025	<0.0025	<0.0025	<0.1	<0.05	<0.05	<0.01	<0.01
S21-040002	<0.0025	<0.0025	<0.0025	<0.0025	<0.1	<0.05	<0.05	<0.01	<0.01
S21-040003	<0.0025	<0.0025	<0.0025	<0.0025	<0.1	<0.05	<0.05	<0.01	<0.01
S21-040011	<0.0025	<0.0025	<0.0025	<0.0025	<0.1	<0.05	<0.05	<0.01	<0.01
S21-040012	<0.0025	<0.0025	<0.0025	<0.0025	<0.1	<0.05	<0.05	<0.01	<0.01
S21-040013	<0.0025	<0.0025	<0.0025	<0.0025	<0.1	0.090	<0.05	<0.01	<0.01
S21-040016	<0.0025	<0.0025	<0.0025	<0.0025	<0.1	<0.05	<0.05	<0.01	<0.01
S21-040017	<0.0025	<0.0025	<0.0025	<0.0025	<0.1	<0.05	<0.05	<0.01	<0.01

*soya infant formula analysed as liquid prepared 25g in 250ml = 0.1g/ml powder
** Highlighted cells not corrected for recovery

Table 13. Contd. Mycotoxin results dairy alternative products using 11+ method

	AFB1	AFB2	AFG1	AFG2	FB1	FB2	FB3	OTA	STG**
Sample number	Concentration, corrected for recovery, µg/kg								
S21-040019	<0.0025	<0.0025	<0.0025	<0.0025	<0.1	<0.05	<0.05	<0.01	<0.01
S21-040020	<0.0025	<0.0025	<0.0025	<0.0025	<0.1	<0.05	<0.05	<0.01	<0.01
S21-040021	<0.0025	<0.0025	<0.0025	<0.0025	<0.10 (0.054q)	<0.05	<0.05	<0.01	<0.01
S21-040023*	<0.0025	<0.0025	<0.0025	<0.0025	<0.1	<0.05	<0.05	<0.01	<0.01
S21-040025	0.005	<0.0025	<0.0025	<0.0025	0.148	<0.05	<0.05	<0.01	<0.01
S21-040027	<0.0025	<0.0025	<0.0025	<0.0025	<0.1	<0.05	<0.05	<0.01	<0.01
S21-040028	0.004	<0.0025	<0.0025	<0.0025	<0.1	<0.05	<0.05	<0.01	<0.01
S21-040029	<0.0025	<0.0025	<0.0025	<0.0025	<0.1	<0.05	<0.05	<0.01	<0.01
S21-040030	<0.0025	<0.0025	<0.0025	<0.0025	<0.1	<0.05	<0.05	<0.01	<0.01
S21-040031	0.003	<0.0025	<0.0025	<0.0025	<0.1	<0.05	<0.05	<0.01	<0.01
S21-040032	0.009	<0.0025	<0.0025	<0.0025	<0.1	<0.05	<0.05	<0.01	<0.01
S21-040008	<0.0025	<0.0025	<0.0025	<0.0025	<0.1	<0.05	<0.05	<0.01	<0.01
Average recovery, % (n=4)	64	67	64	68	75	79	78	70	24

*soya infant formula analysed as liquid prepared 25g in 250ml = 0.1g/ml powder
** Highlighted cells not corrected for recovery

Table 14. Oat product results using accredited multi mycotoxin method - In-house method FSG 818.

	15-AcDON	3-AcDON	DON	NIV	DON-3-G	T-2	HT-2	T-2 a3-G	ZON
Sample Number	Concentration, corrected for recovery, (µg/kg)								
S21-039992	<20	<10	<10 (4.9q)	<50	<10	<10	<10	<10	<2.5
S21-039994	<20	<10	15.6	<50	<10 (6.6q)	<10	<10 (2.7q)	<10	<2.5
S21-039998	<20	<10	27.2	<50	35.0	<10	<10 (5.1q)	<10	<2.5
S21-039999	<20	<10	37.9	<50	28.8	<10	16.4	<10	<2.5
S21-040000	<20	47.5	355	<50	190	10.4	62.8	<10 (2.2q)	7.3
S21-040004	Fail	Fail	Fail	Fail	Fail	Fail	Fail	Fail	Fail
S21-040005	<20	<10	<10	<50	<10	<10	<10 (1.4q)	<10	6.7
S21-040006	<20	<10	<10	<50	<10	<10	<10	<10	<2.5
S21-040007	<20	<10	<10	<50	<10	<10	<10	<10	<2.5
S21-040009	<20	<10	68.8	<50	20.7	<10 (8.9q)	24.1	<10 (6.3q)	<2.5
S21-040010	<20	<10	<10	<50	<10	<10	<10	<10	<2.5
S21-040014	<20	10.3	86.9	<50	62.1	<10	24.6	<10	<2.5
S21-040015	<20	<10	<10 (4.9q)	<50	<10	<10	<10	<10	<2.5
S21-040022	Fail	Fail	Fail	Fail	Fail	Fail	Fail	Fail	Fail
S21-040024	54.1	<10	<10	<50	<10	<10	<10	<10	<2.5
S21-040026	<20	<10	10.1	<50	<10 (5.0q)	<10	<10	<10	<2.5
S21-040033	<20	<10	15.6	<50	<10 (8.7q)	<10	<10 (9.4q)	<10	<2.5
Recovery % (n=4)	104	91	114	89	86	80	81	49*	44*

*Recovery outside acceptable range

Samples also analysed for:

Diacetoxyscirpenol (DAS), fusarenon X (FUSX), neosolaniol (NEO) – no residues were detected above the LOQ of 10 µg/kg for each toxin.

α-zearalenol, β-zearalenol – no residues were detected above the LOQ of 2.5 µg/kg, and zearalenone-14-glucoside, α-zearalenol-glucoside and β-zearalenol-glucoside - no residues were detected above the LOQ of 5 µg/kg.

Table 15. Expanded Measurement Uncertainty values, Sum T-2 and HT-2 results

Sample	Method used	Expanded MU* (%)	Result	MU	Lower limit	Upper limit
		%	µg/kg			
S21-040000	FSG 818	20.2	73.2	16.1	57.1**	89.3
S21-040000	11+	30	56.1	16.8	39.3	72.9
S21-040022	11+	30	23.9	7.17	16.7	31.1

*Expanded MU, with coverage factor of 2, gives 95% confidence interval
** Lower limit exceeds the proposed limit of 50 µg/kg for sum Ht-2 and T-2 toxins

Table 16. Results of 3-MCPD and glycidyl esters in coconut drinks

	3-MCPD	2-MCPD	3-MBPD
LIMS Number	µg/kg, corrected for recovery		
S21-039995	<5	<5	<5
S21-040001	<5	<5	<5
S21-040011	<5	<5	<5
S21-040016	<5	<5	<5
S21-040032	<5	<5	<5
Average spike*	4.8	4.8	4.2
std dev	0.4	2.1	0.4
cv %	7.3	44.2	9.4
recovery %	97	97	83

*spiked at 5 µg/kg each analyte, n=6.

Table 17. Quality control results for 3MCPD analysis

	3MCPD	2MCPD	3MBPD
	µg/kg, corrected for recovery		
IHR 2651	122.2	303.5	138.5
Lower acceptable value	61	188	66
Upper acceptable value	157	420	176

Table 18. Results of isoflavone analyses of soya products

		Daidzien	Daidzin	Genistein	Genistin	Glycitein	Glycitin	Total Isoflavones
LIMS Number	**Sample type**	Average Concentration (n=2, mg/L, or mg/kg), corrected for recovery						
S21-040003	Soya drinks (unsweetened)	6.5	63.4	6.4	283.7	<0.06	16.6	377
S21-039997	Soya drinks (unsweetened)	2.7	121	2.3	362.1	<0.06	<0.06	488
S21-040029	Soya drinks (unsweetened)	1.3	52.2	2.4	238.3	<0.06	<0.06	294
S21-040025	Soya drinks (unsweetened)	3.1	114	4.1	303.9	<0.06	14.9	440
S21-040012	Soya drinks (unsweetened)	1.5	47.3	1.5	185.2	<0.06	7.0	243
S21-040030	Soya drinks (unsweetened)	1.6	77	2.0	132.7	<0.06	<0.06	213
S21-040013	Soya drinks (unsweetened)	<0.06	58	1.3	257.1	<0.06	<0.06	316
S21-040021	Soya drinks (unsweetened)	1.7	43.9	3.3	249.5	<0.06	<0.06	298
S21-039993	Soya formula	11	40.9	18.2	263.7	<0.6	6.1	340
S21-040023	Soya formula	13.5	54.7	16.8	267.7	1.6	14.5	369
Spike 1		84	90	78	102	90	90	
Spike 2		102	102	90	108	96	96	
Average rec (%)		93	96	84	105	93	93	

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Table 19. *Fusarium* Mycotoxin results non-dairy alternative products using multi mycotoxin method - In-house method FSG 818.

		15-AcDON	3-AcDON	DON	NIV	DON-3-G	T-2	HT-2	T-2 a3-G	ZON
Sample number	**Sample description**	Concentration, corrected for recovery, (µg/kg)								
S21-041122	Unsweetened Oat Drink	<20	<10	<10	<50	<10	<10	<10	<10	<2.5
S21-041123	Oat Barista	<20	<10	<10	<50	<10	<10	<10	<10	<2.5
S21-041124	Oat Milk	<20	<10	<10	<50	<10	<10	<10	<10	<2.5
S21-041125	Oat Milk	<20	<10	<10	<50	<10	<10	<10	<10	<2.5

		α-ZOL	β-ZOL	FUSX	DAS	NEO	α-ZOL-G	β-ZOL-G	ZON-G
Sample number	**Sample description**	Concentration, corrected for recovery, (µg/kg)							
S21-041122	Oat Barista	<2.5	<2.5	<10	<10	<10	<5	<5	<5
S21-041123	Oat Milk	<2.5	<2.5	<10	<10	<10	<5	<5	<5
S21-041124	Oat Milk	<2.5	<2.5	<10	<10	<10	<5	<5	<5
S21-041125	Unsweetened Oat Drink	<2.5	<2.5	<10	<10	<10	<5	<5	<5

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