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Monitoring the Presence of Ergot Alkaloids in Cereals and a Study of a Possible Relationship between Occurrence of Sclerotia Content and Levels of Ergot Alkaloids

Rep AC/132779/1

Food Standards Agency

Final Report

Project Number FS516009

12 November 2014

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Contents

Summary

In total 209 samples of cereal grains (wheat, barley, oats and rye) were analysed for the six major ergot alkaloids and epimers. Overall the level of ergot alkaloid contamination was low. The highest frequency of contamination (64%) and highest total ergot alkaloid levels (370 µg/kg) were determined in rye samples.

The effectiveness of reducing ergot alkaloid content by the removal of sclerotia was also investigated. The cleaning processes studied appear to have reduced the levels of alkaloids significantly; however only a relatively small number of samples have been analysed in this study.

Sponser

The work in this report was funded by the UK Food Standards Agency (Project Number FS516009).

Background

Ergot alkaloids (EAs) are mycotoxins produced by fungi of the Claviceps genus; the most notable in Europe being Claviceps purpurea which parasitise the seed heads of living plants at the time of flowering.

These fungal infections are most prevalent in rye and triticale that have open florets but also wheat and other small grains are potential hosts. The fungus replaces the developing grain or seed with the alkaloid-containing wintering body, known as ergot, ergot body or sclerotium. The sclerotia are harvested together with the cereals or grass and can thus lead to contamination of cereal-based food and feed products with ergot alkaloids, ingestion of which can cause ergotism in humans and animals. The main ergot alkaloids produced by the Claviceps species which are contained in the sclerotia are ergometrine, ergotamine, ergosine, ergocristine, ergocryptine, ergocornine and their corresponding –inine epimers.

Objective

Following a request from the European Commission, EFSA's Panel on Contaminants in the Food Chain (CONTAM Panel), in June 2012, delivered a scientific opinion on the risks to human and animal health related to the presence of EAs in food and feed. The panel recommended, among other things, that 'collection of analytical data on occurrence of EAs in relevant food and feed commodities should continue.' This proposed work is in response to this recommendation. Discussions on managing the risks associated with the presence of EAs in food and feed are taking place at the European Commission and currently negotiations are ongoing. Data generated from this work will be used to feed into those negotiations.

Current controls put in place by millers and grain processors include processes such as physical separation/cleaning through visual inspection and separation using optical sorters to remove discoloured and mis-shapened grains.

The industry has almost a zero tolerance for presence of ergot sclerotia in grains such as wheat, oat and barley i.e. any visible sclerotia on inspection will lead to the entire lot being rejected. Removal of the sclerotia leads to a considerable reduction in the levels of ergot alkaloids in the grain. However, it is not possible to detect the presence of dust and smaller particles resulting from breakage of the sclerotia and therefore ergot alkaloids could be present.

There are six main ergot alkaloids and corresponding epimers (12 in total) and the profile and concentration of individual alkaloids varies considerably in different grains and batches of grain. The toxicity of the individual alkaloids also varies. This study proposes to look at the ergot alkaloid content of grains from the 2013 harvest and the effectiveness of reducing ergot alkaloid content from contaminated samples by removing sclerotia by visual and automated means.

Methodology

Safety

All procedures described in this report were conducted at Campden BRI**.** All laboratory workers have been trained for work with ergot alkaloids. Control of Substances Hazardous to Health (COSHH); risk assessment of ergot alkaloid assay was prepared prior to undertaking specific jobs.

Materials and Chemicals

All reagents were Analytical (AR) grade. All solvents were of LC/MS grade purchased from Fisher Scientific. Primary Secondary Amine was purchased from Agilent .

Mycotoxin Standards

Ergot Alkaloid and epimer standards were purchased from Romer Labs

Samples

Sample Collection and Preparation

A total of 197 samples were gratefully received from members of Nabim, MAGB and AIC, in addition to samples received from Morning Foods Ltd. Sixteen of the samples received were 'rejected' samples, meaning they were thought to contain high levels of ergot alkaloids and therefore would have been directed away from the human food chain. Twelve of the thirty six rejected samples were analysed before and after undergoing a cleaning process. A total of 209 analyses were therefore undertaken. All samples were delivered to Campden BRI, at ambient temperature and stored at –20°C until required for analysis.

Grain	Number of samples
Wheat	10
Wheat -Rejected	20
Wheat For Feed	65
Wheat For Feed - Rejected	1
Oats	8
Oats – Rejected	1
Oats – Cleaned	1
Barley	29
Barley – Rejected	14
Rye Cleaned	42
Pre-Cleaned Rye	6

Preparation of Samples

All samples in this survey were milled and thoroughly mixed to ensure homogeneity prior to analysis. After homogenisation the samples were stored in a freezer at –

20°C. Samples were allowed to defrost to ambient temperature prior to analysis and any remaining sample returned to –20°C immediately after analysis.

Laboratory cleaning of samples

Thirteen grain samples rejected at mill for suspected high levels of ergot contamination were analysed before and after laboratory cleaning to determine effectiveness of sclerotia removal in reducing ergot alkaloid content. Rejected samples were divided into two, one half was analysed without further clean up whilst the second was sorted visibly within the laboratory to remove sclerotia and discoloured grains.

Analytical Methodology

The ergot alkaloid and epimers (ergocristine, ergotamine, ergocornine, ergosine, ergocryptine, ergometrine, ergocristinine, ergotaminine, ergocorninine, ergosinine, ergocryptinine and ergometrinine) were determined by LC/MS/MS.

Ground samples (5g) were extracted with 25 ml acetonitrile/ammonium carbonate buffer (84:16) by shaking for 30 minutes on an orbital shaker. A 2ml aliquot of the extract was vortexed with 100mg of PSA for 45 seconds. The filtered extract was analysed by LC/MS/MS. Quantative analysis was performed using matrix-matched calibration standards.

In total 209 samples of cereal grains (wheat, barley, oats and rye) were analysed for the six major ergot alkaloids and epimers. Samples of wheat and barley rejected at intake for suspected unacceptable levels of ergot contamination were divided into two equal portions. One half was analysed without any clean up, whilst the second half was analysed after sclerotia had been removed following visual examination. Selected rye and oat samples were cleaned by an industrial ergot sclerotia removal process. Samples were analysed pre and post cleaning. The rye samples had been cleaned by a three stage industrial process which culminated in discoloured and irregular matter being removed by an optical sorter. An optical sorting process was also used to clean the oat samples. Eleven rejected barley samples were analysed before and after sclerotia removal. One rejected wheat sample (grain for feed) was similarly split into two and analysed before and after sclerotia removal. Six samples of rye were taken prior to, and after industrial cleaning to remove sclerotia. One oat sample (pre and post cleaning- cleaning technique unknown) was received for analysis.

Recovery and Limit of Quantification

All analyses were conducted with a spiked sample, i.e. to each sample matrix a known amount of toxin was added prior to extraction, clean-up and LC/MS/MS determination for each batch of 1-10 samples. These results were used to assess recovery and all reported results were corrected using the values obtained. Recoveries in the range 60-120% were considered acceptable. The spiking level was 50µg/kg. The limit of Quantification for all alkaloids was 1 µg/kg.

Uncertainty of Measurement

Uncertainty of measurement calculations were calculated using in-house data. The expanded measurement of uncertainty was calculated using a standard coverage of 2.

Table 2: Uncertainty of Measurement

Mycotoxin	**Uncertainty**
Ergocornine	x µg/kg ± 10%
Ergocristine	x µg/kg ± 9%
Ergocryptine	x µg/kg ± 10%
Ergometrine	x µg/kg ± 31%
Ergosine	x µg/kg ± 14%
Ergotamine	x µg/kg ± 11%
Ergocorninine	x µg/kg ± 19%
Ergocristinine	x µg/kg ± 17%
Ergocryptinine	x µg/kg ± 15%
Ergometrinine	x µg/kg ± 32%
Ergosinine	x µg/kg ± 18%
Ergotaminine	x µg/kg ± 22%

Survey results

A total of 209 samples comprised of wheat, rye, barley and oat samples destined for either human or animal consumption were analysed. Also included were samples rejected at intake for possible ergot contamination, and samples that had been 'cleaned' (visible removal of any apparent sclerotia) within the laboratory. These samples would not have entered the food chain.

The majority of samples (excluding rejected samples) did not have any ergot alkaloids (or epimers) above the LOQ of 1µg/kg as shown in Table 1.

Table 1 below summarises the results of this survey (excluding rejected samples):

Summary of Ergot Alkaloid and Epimer Occurrence in Products Analysed	**Number of Samples Analysed**	**Sample Destination**	**Number (%) of samples in which one of more ergot alkaloid and epimers were detected at or above the LOD (1 µg/kg)**
Wheat	10 *	Human Consumption	2 (20%)
Rye	36	Human Consumption	23 (64%)
Barley	29	Human Consumption	2 (7%)
Oats	8	Human Consumption	3 (38%)
Wheat	65	Grain For Feed	12 (18%)

A summary of results for the different grain types (excluding rejected samples) are shown in tables 3 to 7.

Detailed results for all samples are shown in tables 8-17.

When calculating the total ergot alkaloid content of each sample, ergot alkaloids, residues determined at <LOQ (<1 µg/kg) were assigned a value of zero to calculate total ergot alkaloid content (LOQ = 1 µg/kg).

Table 3: Rye Samples

Alkaloid	Total Number of samples analysed	Number of samples in which mycotoxin was determined ≥ (LOQ)	Range of results above LOQ (µg/kg)
Ergocornine	36	4	1-28
Ergotamine	36	15	1-256
Ergosine	36	13	1-27
Ergocryptine	36	7	1-20
Ergocristine	36	14	1-52
Ergometrine	36	4	2-11
Ergocorninine	36	3	2-9
Ergotaminine	36	10	1-67
Ergosinine	36	9	1-8
Ergocryptinine	36	8	1-11
Ergocristinine	36	10	1-5
Ergometrinine	36	1	2

Ta**ble 4: Oat Samples**

Mycotoxin	Total Number of samples analysed	Number of samples in which mycotoxin was determined ≥ (LOQ)	Range of results above LOQ (µg/kg)
Ergocornine	8	1	3
Ergotamine	8	2	2-48
Ergosine	8	3	4-31
Ergocryptine	8	2	2-5
Ergocristine	8	1	2
Ergometrine	8	1	12
Ergocorninine	8	1	4
Ergotaminine	8	2	2-43
Ergosinine	8	2	3-45
Ergocryptinine	8	2	2-3
Ergocristinine	8	1	2
Ergometrinine	8	1	1

Table 5: Wheat Samples

Mycotoxin	Total Number of samples analysed	Number of samples in which mycotoxin was determined ≥ (LOQ)	Range of results above LOQ (µg/kg)
Ergocornine	10	0	-
Ergotamine	10	1	2
Ergosine	10	1	3
Ergocryptine	10	1	12
Ergocristine	10	2	1-10
Ergometrine	10	0	-
Ergocorninine	10	0	-
Ergotaminine	10	1	18
Ergosinine	10	0	-
Ergocryptinine	10	1	3
Ergocristinine	10	0	-
Ergometrinine	10	0	-

Table 6: Barley Samples

Mycotoxin	Total Number of samples analysed	Number of samples in which mycotoxin was determined ≥ (LOQ)	Range of results above LOQ (µg/kg)
Ergocornine	29	1	10
Ergotamine	29	1	6
Ergosine	29	2	4-14
Ergocryptine	29	1	6
Ergocristine	29	0	-
Ergometrine	29	0	-
Ergocorninine	29	0	-
Ergotaminine	29	0	-
Ergosinine	29	1	1
Ergocryptinine	29	0	-
Ergocristinine	29	0	-
Ergometrinine	29	0	-

Table 7: Wheat (grain for feed) Samples

Mycotoxin	Total Number of samples analysed	Number of samples in which mycotoxin was determined ≥ (LOQ)	Range of results above LOQ (µg/kg)
Ergocornine	65	5	1-20
Ergotamine	65	7	3-83
Ergosine	65	5	5-93
Ergocryptine	65	8	2-15
Ergocristine	65	5	1-181
Ergometrine	65	1	3
Ergocorninine	65	5	1-5
Ergotaminine	65	5	2-19
Ergosinine	65	7	1-26
Ergocryptinine	65	4	2-4
Ergocristinine	65	4	1-40
Ergometrinine	65	0	-

Table 8: Ergot Alkaloids (µg/kg) in Rye Samples

Sample	Ergocornine	Ergotamine	Ergosine	Ergocryptine	Ergocristine	Ergometrine	Ergocorninine	Ergotaminine	Ergosinine	Ergocryptinine	Ergocristinine	Ergometrinine	Tota alkaloidsl
1	(<LOD)	2	1	(<LOD)	5	(<LOD)	(<LOD)	1	(<LOD)	(<LOD)	3	(<LOD)	12
2	(<LOD)	256	5	16	5	11	(<LOD)	67	1	5	1	2	369
3	11	70	13	5	7	2	2	18	3	1	1	(<LOD)	133
4	(<LOD)	4	1	2	52	3	(<LOD)	1	(<LOD)	(<LOD)	5	(<LOD)	68
5	(<LOD)	1	3	(<LOD)	3	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	1	(<LOD)	8
6	(<LOD)	(<LOD)	4	1	(<LOD)	(<LOD)	(<LOD)	(<LOD)	1	(<LOD)	(<LOD)	(<LOD)	6
7	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	1	(<LOD)	1
8	(<LOD)	1	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	1	(<LOD)	1	(<LOD)	(<LOD)	3
9	(<LOD)	3	(<LOD)	(<LOD)	1	(<LOD)	(<LOD)	2	(<LOD)	(<LOD)	2	(<LOD)	8
10	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
11	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
12	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
13	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
14	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
15	(<LOD)	6	4	(<LOD)	(<LOD)	(<LOD)	(<LOD)	8	2	(<LOD)	(<LOD)	(<LOD)	20
16	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
17	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
18	(<LOD)	(<LOD)	(<LOD)	1	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	3	(<LOD)	(<LOD)	4
19	(<LOD)	(<LOD)	(<LOD)	(<LOD)	4	(<LOD)	(<LOD)	1	1	1	(<LOD)	(<LOD)	7
20	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
21	(<LOD)	(<LOD)	1	(<LOD)	2	(<LOD)	(<LOD)	(<LOD)	1	(<LOD)	2	(<LOD)	610
22	(<LOD)	1	1	(<LOD)	1	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	3
23	(<LOD)	81	5	(<LOD)	5	2	(<LOD)	13	1	(<LOD)	2	(<LOD)	109
24	(<LOD)	(<LOD)	5	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	5
25	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
26	(<LOD)	4	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	4
27	(<LOD)	1	(<LOD)	(<LOD)	3	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	4
28	(<LOD)	4	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	1	(<LOD)	(<LOD)	5
29	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
30	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
31	28	(<LOD)	27	20	(<LOD)	(<LOD)	9	(<LOD)	8	11	(<LOD)	(<LOD)	103
32	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
33	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
34	1	3	(<LOD)	(<LOD)	1	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
35	(<LOD)	(<LOD)	(<LOD)	(<LOD)	1	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	1
36	9	3	3	7	2	(<LOD)	2	1	1	4	(<LOD)	(<LOD)	32

Table 9: Ergot Alkaloids (µg/kg) in Rye Pre-cleaned and Cleaned Samples

	Ergocornine	Ergotamine	Ergosine	Ergocryptine	Ergocristine	Ergometrine	Ergocorninine	Ergotaminine	Ergosinine	Ergocryptinine	Ergocristinine	Ergometrinine	Total
Pre-clean	2	4	24	7	48	(<LOD)	(<LOD)	18	6	9	9	(<LOD)	127
Pre-clean	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
Pre-clean	(<LOD)	23	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	3	(<LOD)	(<LOD)	(<LOD)	26
Pre-clean	(<LOD)	(<LOD)	8	(<LOD)	(<LOD)	2	(<LOD)	75	(<LOD)	(<LOD)	(<LOD)	(<LOD)	85
Pre-clean	(<LOD)	(<LOD)	(<LOD)	(<LOD)	44	59	(<LOD)	(<LOD)	4	(<LOD)	17	16	140
Pre-clean	6	(<LOD)	24	15	(<LOD)	(<LOD)	1	(<LOD)	(<LOD)	9	(<LOD)	(<LOD)	55
Cleaned	(<LOD)	1	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	1
Cleaned	1	(<LOD)	(<LOD)	1	1	1	1	1	(<LOD)	1	1	(<LOD)	8
Cleaned	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
Cleaned	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
Cleaned	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
Cleaned	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0

*****Samples cleaned by industrial process (Please note: these samples were taken from the same batch in order to examine differences between cleaned and pre-cleaned results and were therefore analysed twice).

Table 10: Ergot Alkaloids (µg/kg) in Oat Samples

Sample	Ergocornine	Ergotamine	Ergosine	Ergocryptine	Ergocristine	Ergometrine	Ergocorninine	Ergotaminine	Ergosinine	Ergocryptinine	Ergocristinine	Ergometrinine	Total
1	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
2	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
3	(<LOD)	48	8	5	(<LOD)	(<LOD)	(<LOD)	43	45	3	(<LOD)	(<LOD)	152
4	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	06
5	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
6	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
7	(<LOD)	(<LOD)	31	(<LOD)	(<LOD)	12	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	1	44
8	3	2	4	2	2	(<LOD)	4	2	3	2	2	(<LOD)	26

Table 11: Ergot Alkaloids (µg/kg) in Rejected Oat Sample

	Ergocornine	Ergotamine	Ergosine	Ergocryptine	Ergocristine	Ergometrine	Ergocorninine	Ergotaminine	Ergosinine	Ergocryptinine	Ergocristinine	Ergometrinine	Total
Pre-clean	155	223	341	129	493	16	108	213	219	87	516	1	2501
*Cleaned	20	34	20	12	20	(<LOD)	13	56	17	9	18	(<LOD)	219

*****Sample cleaned by industrial process

Table 12: Ergot Alkaloids (µg/kg) in Wheat Samples

	Ergocornine	Ergotamine	Ergosine	Ergocryptine	Ergocristine	Ergometrine	Ergocorninine	Ergotaminine	Ergosinine	Ergocryptinine	Ergocristinine	Ergometrinine	Total
1	<LOD	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
2	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
3	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
4	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
5	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
6	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
7	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
8	(<LOD)	(<LOD)	(<LOD)	(<LOD)	1	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
9	(<LOD)	2	3	12	10	(<LOD)	(<LOD)	18	(<LOD)	3	(<LOD)	(<LOD)	48
10	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0	6

Table 13: Ergot Alkaloids (µg/kg) in Rejected Wheat Samples

	Ergocornine	Ergotamine	Ergosine	Ergocryptine	Ergocristine	Ergometrine	Ergocorninine	Ergotaminine	Ergosinine	Ergocryptinine	Ergocristinine	Ergometrinine	Total
1	259	429	617	141	716	10	98	147	184	43	247	3	2894
2	159	4	168	70	7	4	107	2	62	36	3	3	625
3	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
4	12	61	90	10	82	(<LOD)	3	20	27	3	27	(<LOD)	335
5	27	54	199	20	197	(<LOD)	12	18	55	7	63	(<LOD)	652
6	68	204	548	43	760	31	29	68	181	16	253	(<LOD)	2201
7	(<LOD)	1	2	(<LOD)	7	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	10
8	(<LOD)	(<LOD)	3	(<LOD)	1	(<LOD)	(<LOD)	(<LOD)	1	(<LOD)	(<LOD)	(<LOD)	4
9	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
10	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
11	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
12	(<LOD)	2	2	(<LOD)	4	(<LOD)	(<LOD)	(<LOD)	1	(<LOD)	1	(<LOD)	10
13	(<LOD)	2	(<LOD)	(<LOD)	6	(<LOD)	(<LOD)	1	(<LOD)	(<LOD)	4	(<LOD)	13
14	13	(<LOD)	21	7	(<LOD)	(<LOD)	4	(<LOD)	(<LOD)	2	(<LOD)	(<LOD)	47
15	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
16	(<LOD)	(<LOD)	1	(<LOD)	3	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	4
17	27	11	41	21	31	(<LOD)	13	5	16	8	27	(<LOD)	200
18	(<LOD)	1	16	(<LOD)	4	(<LOD)	(<LOD)	(<LOD)	6	(<LOD)	1	(<LOD)	28

19	(<LOD)	64	100	1	153	(<LOD)	(<LOD)	23	30	(<LOD)	44	(<LOD)	415
20	(<LOD)	59	1	2	5	(<LOD)	(<LOD)	29	(<LOD)	1	2	(<LOD)	99

Table 14: Ergot Alkaloids (µg/kg) in Barley Samples

	Ergocornine	Ergotamine	Ergosine	Ergocryptine	Ergocristine	Ergometrine	Ergocorninine	Ergotaminine	Ergosinine	Ergocryptinine	Ergocristinine	Ergometrinine	Total
1	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
2	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
3	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
4	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
5	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
6	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
7	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
8	10	(<LOD)	14	6	(<LOD)	(<LOD)	(<LOD)	(<LOD)	1	(<LOD)	(<LOD)	(<LOD)	31
9	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
10	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
11	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
12	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
13	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
14	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
15	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
16	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
17	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
18	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
19	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
20	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
21	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
22	(<LOD)	6	4	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	10
23	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
24	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
25	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
26	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
27	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
28	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
29	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0

Table 15: Ergot Alkaloids (µg/kg) in Rejected Barley Samples

		Ergocornine	Ergotamine	Ergosine	Ergocryptine	Ergocristine	Ergometrine	Ergocorninine	Ergotaminine	Ergosinine	Ergocryptinine	Ergocristinine	Ergometrinine	Total
1_1		(<LOD)	55	(<LOD)	28	(<LOD)	(<LOD)	(<LOD)	21	(<LOD)	12	(<LOD)	(<LOD)	116
1_2	C	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
2_1		6	5	25	3	11	(<LOD)	1	1	6	1	4	(<LOD)	63
2_2	C	(<LOD)	3	3	(<LOD)	16	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	2	(<LOD)	24
3_1		147	318	364	87	316	13	36	67	87	22	99	1	1557
3_2	C	52	48	96	38	133	3	8	5	14	16	15	(<LOD)	428
4_1		(<LOD)	(<LOD)	382	1	1	(<LOD)	(<LOD)	64	(<LOD)	(<LOD)	(<LOD)	(<LOD)	448
4_2	C	(<LOD)	(<LOD)	(<LOD)	(<LOD)	5	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	5
5_1		(<LOD)	3	2	(<LOD)	1	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	6
5_2	C	(<LOD)	(<LOD)	7	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	7
6		10	11	26	7	61	(<LOD)	1	1	4	1	4	(<LOD)	126
7_1		71	9	80	29	75	(<LOD)	6	1	12	4	5	(<LOD)	292
7_2	C	(<LOD)	4	20	(<LOD)	13	(<LOD)	(<LOD)	(<LOD)	2	(<LOD)	(<LOD)	(<LOD)	39
8_1		333	1141	761	245	1833	29	78	229	279	42	255	29	5254
8_2	C	1	5	67	1	(<LOD)	(<LOD)	(<LOD)	(<LOD)	14	(<LOD)	(<LOD)	(<LOD)	88
9_1		(<LOD)	(<LOD)	(<LOD)	(<LOD)	1	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	1
9_2	C	4	5	18	2	18	(<LOD)	(<LOD)	(<LOD)	2	(<LOD)	1	(<LOD)	50
10		5	2	3	2	9	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	1	(<LOD)	22
11_1		(<LOD)	4	(<LOD)	5	23	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	1	(<LOD)	33
11_2	C	4	5	18	2	19	(<LOD)	(<LOD)	(<LOD)	2	(<LOD)	1	(<LOD)	51
12		(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
13_1		107	197	269	71	567	19	17	26	58	15	60	6	1412
13_2	C	35	133	264	24	235	11	11	22	58	8	38	2	841
14_1		66	283	181	61	274	8	7	32	29	7	19	(<LOD)	967
14_2	C	33	142	132	16	294	1	4	18	19	3	21	(<LOD)	683

C – Sample cleaned in laboratory

Table 16: Ergot Alkaloids (µg/kg) in Wheat (grain for feed) Samples

	Ergocornine	Ergotamine	Ergosine	Ergocryptine	Ergocristine	Ergometrine	Ergocorninine	Ergotaminine	Ergosinine	Ergocryptinine	Ergocristinine	Ergometrinine	Total
1	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
2	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
3	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
4	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
5	1	6	5	(<LOD)	15	(<LOD)	1	3	2	(<LOD)	6	(<LOD)	39
6	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
7	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
8	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
9	(<LOD)	83	45	(<LOD)	(<LOD)	(<LOD)	(<LOD)	19	7	(<LOD)	(<LOD)	(<LOD)	154
10	11	3	12	4	8	3	3	(<LOD)	3	(<LOD)	1	(<LOD)	48
11	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
12	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
13	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
14	20	(<LOD)	21	10	(<LOD)	(<LOD)	5	(<LOD)	6	4	(<LOD)	(<LOD)	66
15	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
16	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
17	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
18	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
19	(<LOD)	11	(<LOD)	2	(<LOD)	(<LOD)	(<LOD)	2	(<LOD)	(<LOD)	(<LOD)	(<LOD)	15
20	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
21	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
22	(<LOD)	39	93	13	181	(<LOD)	4	13	26	4	40	(<LOD)	413
23	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
24	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
25	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
26	(<LOD)	(<LOD)	(<LOD)	(<LOD)	1	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	1
27	(<LOD)	(<LOD)	(<LOD)	4	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	4
28	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
29	(<LOD)	77	(<LOD)	15	6	(<LOD)	(<LOD)	14	(<LOD)	3	1	(<LOD)	116
30	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
31	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0

32	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
33	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
34	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
35	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
36	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
37	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
38	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
39	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
40	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
41	7	(<LOD)	(<LOD)	3	(<LOD)	(<LOD)	2	(<LOD)	3	2	(<LOD)	(<LOD)	17
42	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
43	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
44	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
45	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
46	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
47	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
48	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
49	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
50	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
51	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
52	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
53	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
54	(<LOD)	4	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	1	(<LOD)	(<LOD)	(<LOD)	5
55	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
56	2	(<LOD)	(<LOD)	2	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	4
57	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
58	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
59	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
60	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
61	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
62	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
63	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
64	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0
65	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	0

Table 17: Ergot Alkaloid (µg/kg) in Rejected Wheat (grain for feed) Sample

		Ergocornine	Ergotamine	Ergosine	Ergocryptine	Ergocristine	Ergometrine	Ergocorninine	Ergotaminine	Ergosinine	Ergocryptinine	Ergocristinine	Ergometrinine	Total
1		4610	3305	4166	3048	464	188	1425	602	1486	1027	105	31	20457
	C	(<LOD)	(<LOD)	1	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	(<LOD)	1

C – Sample cleaned in laboratory

Statistical Analysis

Samples were grouped into cereal type and categories as shown in Table 18. The geometric means of the total alkaloid concentration and the numbers of samples in each group are shown in Table 19.

Table 18: Grouping of samples: text description

Cereal type	Count	Category
Barley	28	A
Barley Rejected	14	B
Barley Rejected-Cleaned	11	C
Oats	8	A
Oats Rejected	1	B
Oats Rejected Cleaned By Supplier	1	C
Rye	36	A
Rye-Precleaned	6	B
Rye-Cleaned	6	C
Wheat	75	A
Wheat Rejected	21	B
Wheat Rejected –Cleaned	1	C

Table 19: Geometrics mean of Total alkaloid concentration (µg/kg):
mean (µg/kg) (number of samples); '<1' treated as 1/√2

Grain	**Category**			
	A	B	C	All
Barley	1 (28)	62 (14)	86 (11)	5 (53)
Oats	4 (8)	2501 (1)	219 (1)	11 (10)
Rye	4 (36)	34 (6)	1 (6)	4 (48)
Wheat	1 (75)	51 (21)	8 (1)	3 (97)
All	2 (147)	55 (42)	12 (19)	4 (208)

As can be seen above, samples were divided into different categories based on their status i.e. category A – the sample type, category B – the rejected samples, category C – the rejected samples which have been cleaned. For barley and wheat there was strong evidence that alkaloid contamination was lower in Category A than in Categories B and C. For oats and rye the evidence for similar differences was not statistically significant; the indications may be artefacts of the small sample numbers.

The evidence for differences between Categories B and C was not statistically significant. Although the sample data suggested that Category C contamination was lower than that of Category B, the data was consistent with no difference or even differences in the other direction. More samples in those categories would be needed to reduce the uncertainty.

Details of the statistical analysis are included in Annex II.

Conclusion

In total 209 samples of cereal grains (wheat, barley, oats and rye) were analysed for the six major ergot alkaloids and epimers.

Overall the levels of the alkaloids found in this survey were low. Excluding data from rejected samples, the highest frequency of contamination and highest levels were determined in rye samples (64% contaminated, 369 µg/kg - highest total ergot level), whilst the lowest frequency of contamination (7%) was determined in barley samples (highest total level of 31 µg/kg).

Samples of wheat and barley rejected at intake for suspected unacceptable levels of ergot contamination were divided into two equal portions. One half was analysed without any clean up, whilst the second half was analysed after sclerotia had been removed following visual examination. Selected rye and oat samples were cleaned by an industrial ergot sclerotia removal process. Samples were analysed pre and post cleaning. The rye samples were cleaned by a three stage industrial process which culminated in discoloured and irregular matter being removed by an optical sorter. An optical sorting process was also used to clean the oat samples. Eleven rejected barley samples were analysed before and after sclerotia removal. Eight of the eleven samples had a lower total ergot alkaloid content after cleaning, with three samples being reduced by 98% or greater. The mean decrease in total ergot alkaloid content in samples showing a reduction after cleaning was 74%.

One rejected wheat sample (grain for feed) was similarly split into two and analysed before and after sclerotia removal. An initial alkaloid residue of 20457µg/kg was reduced to 1µg/kg in the cleaned up sample.

Six samples of rye were taken prior to, and after industrial cleaning to remove sclerotia. Five of the six (83%) pre-cleaned samples contained detectable levels of ergot alkaloids compared to two (33%)of the six samples after cleaning. Total mean ergot alkaloid content dropped from 72 to 2 µg/kg.

One oat sample (pre and post cleaning- cleaning technique unknown) was received for analysis. The initial total alkaloid content of 2501 µg/kg was reduced to 220µg/kg after cleaning.

As can be seen from the above, it appears the cleaning process has significantly reduced the levels of alkaloids, although these are not statistically relevant due to the low number of samples analysed in this work.

ANNEX I: Glossary of Abbreviations

AR	Analytical Reagent
C	Cleaned
COSHH	Control of Substances Hazardous to Health
LC/MS/MS	Liquid Chromatography Tandem Mass Spectrometry
id	Internal Diameter
LOD	Limit of Detection
LOQ	Limit of Quantification
nd	Not Detected
ppb or µg/kg	Parts per Billion or Microgram/Kilogram
ppm or µg/g	Parts per Million or Microgram/Gram
RSD	Relative Standard Deviation
TDI	Tolerable Daily Intake

ANNEX II: Statistical report

Interpretation of workbook contents

Workbook name; [e.g. “ergocornine”] indicates measurand; data taken from worksheet ‘Data Reported’.
Corrected data as of 5 December 2014.
Column contents:

A: “Laboratory sample code (S.01)”; [e.g. “ac/132779/006”] 13-character sample identifier. Unique within a workbook; identifies same sample in different workbooks; may be followed by a two character suffix indicating the same sample at different stages e.g.
ac/132779/131_1 wheat rejected
ac/132779/131_2 wheat rejected-cleaned

N: “Product full text description (S.14)”;

Product full text description	Count
barley	28
barley rejected	14
barley rejected-cleaned	11
oats	8
oats rejected	1
oats rejected cleaned by supplier	1
rye	36
rye-cleaned	6
rye-precleaned	6
wheat	75
wheat rejected	21
wheat rejected-cleaned	1

BI: “Result LOQ (R.15)”; measurand limit of quantification

BL: “Result value (R.18)”; measurand value. Blank means ‘<LOQ’

BT: “Type of result (R.27)”; [“VAL” or “Non Quantified Value (<LOQ)”]; indication of whether BL is value or below BI.

Values were checked to confirm that

- columns A, N, BI were identical across workbooks
- column BI always = 1;
- one-to-one correspondence between BT = “Non Quantified Value (<LOQ)” and BL = blank
- no BL values < 1.

Data pre-treatment

A new worksheet was created holding a single copy of “Laboratory sample code” and of “Product full text description”. Additional columns were created to separate parts of ‘Laboratory sample code’ (‘Code’, ‘Suffix’) and of ‘Product full text description’ (‘Grain’, ‘Category’).

‘Grain’ was taken from the first word of ‘Product full text description’; ‘Category’ was coded as ‘A’, ‘B’ or ‘C’ according to the subsequent text.

Grain	Category		
	A	B	C
barley	[blank]	rejected	rejected-cleaned
oats	[blank]	rejected	rejected cleaned by supplier
rye	[blank]	-precleaned	-cleaned
wheat	[blank]	rejected	rejected-cleaned

“Result value” columns from all worksheets were appended to the new worksheet, blank values corresponding to “Non Quantified Value (<LOQ)” were coded as missing, resulting in the data set described in Table 1.

Table 1: Data as analysed

	Column	Count	Missing	Name
T	C1	208	0	Laboratory sample code
T	C2	208	0	Product full text description
T	C3	208	0	Code
T	C4	208	182	Suffix
T	C5	208	0	Grain
T	C6	208	0	Category
	C7	208	170	ergocornine
	C8	208	177	ergocorninine
	C9	208	146	ergocristine
	C10	208	161	ergocristinine
	C11	208	157	ergocryptine
	C12	208	168	ergocryptinine
	C13	208	187	ergometrine
	C14	208	197	ergometrinine
	C15	208	144	ergosine
	C16	208	159	ergosinine
	C17	208	145	ergotamine
	C18	208	163	ergotaminine

Data analysis

There were very few samples in some groups, Table 2, hampering exploration of some differences.

Table 2: Numbers of samples in each group

		Category			
		A	B	C	All
Grain	barley	28	14	11	53
	oats	8	1	1	10
	rye	36	6	6	48
	wheat	75	21	1	97
	All	147	42	19	208

There were many missing values denoting “<1”, hampering quantification of differences. Accordingly data analysis proceeded in three phases:

- Qualitative: Presence/Absence. Considering only whether alkaloids were detected, that is not “<1”.
- Semi-quantitative: ranks of values. Considering only the rank order of reported values, not their magnitudes. In this phase “<1” ranked lower than any other value.
- Fully quantitative: Considering the values reported. This phase was severely restricted by the large number of results which were not reported as a value, just “<1”.

Qualitative: Presence/Absence

rain and Category within Grain.
Table 3 shows the number of samples in which any of the 12 alkaloids was detected, and binary logistic regression (using R[1]) to test for dependence of contamination (at least one alkaloid detected) on Grain and Category within each Grain. There was statistically significant evidence ($p < 0.001$) that contamination probability depended on both Grain and Category within Grain.

Table 3: Numbers of samples with at least one alkaloid detected: positive samples/total samples

		Category			
		A	B	C	All
Grain	barley	2/28	13/14	10/11	25/53
	oats	3/8	1/1	1/1	5/10
	rye	23/36	5/6	2/6	30/48
	wheat	14/75	16/21	1/1	31/97
	All	42/147	35/42	14/19	91/208

Call: glm(formula = Contam ~ Grain + Category %in% Grain - 1,family = binomial,…)

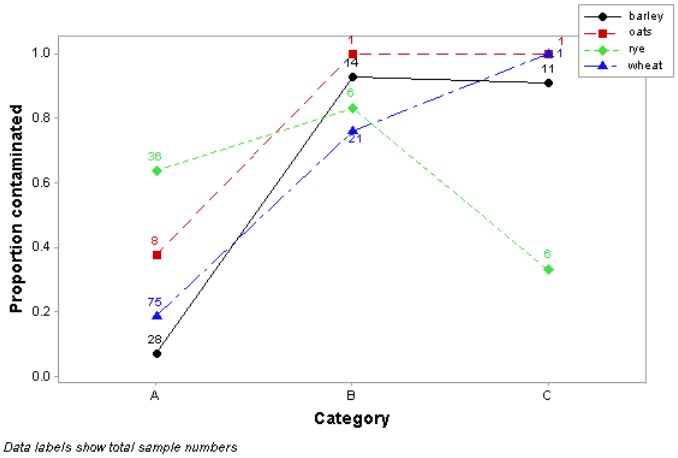
	Df	Sum Sq	Mean Sq	F value	Pr(>F)
Grain	4	42.95	10.737	67.37	< 2e-16 ***
Grain:Category	8	16.81	2.102	13.19	3.56e-15 ***
Residuals	196	31.24	0.159		

Signif. codes: 0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 ‘.’ 0.1 ‘ ’ 1

Figure 1 makes clear that Category A differed from B and C for barley, oats, and wheat; differences between B and C are less clear.

[1] R Core Team (2014). R: A language and environment for statistical computing. R Foundation for Statistical Computing, Vienna, Austria. URL http://www.R-project.org/

Figure 1: Proportions of samples with at least one alkaloid detected



Data labels show total sample numbers

Table 4 shows no statistically significant evidence (p = 0.258) for differences in contamination probability between Categories B and C within Grain.

Table 4: Binary logistic regression for contamination probability; excluding Category A

```
Call:  glm(formula = Contam ~ Grain + Category %in% Grain - 1,
family = binomial, data = subset(…, Category != "A"))

               Df Sum Sq Mean Sq F value Pr(>F)
Grain           4  40.38  10.095  68.472 <2e-16 ***
Grain:Category  4   0.81   0.202   1.368  0.258
Residuals      53   7.81   0.147
---
Signif. codes:  0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 ‘.’ 0.1 ‘ ’ 1
```

Table 5 shows the mean number of the 12 alkaloids which were detected for samples in each group.

Table 5: Mean number of different alkaloids detected (of 12 tested) (mean number/total samples)

		Category			
		A	B	C	All
Grain	barley	0.2/28	7.1/14	5.7/11	3.2/53
	oats	2.4/8	12.0/1	10.0/1	4.1/10
	rye	2.7/36	4.0/6	1.5/6	2.7/48
	wheat	0.8/75	5.6/21	1.0/1	1.9/97
	All	1.3/147	6.0/42	4.4/19	2.5/208

The dependence of number of different alkaloids on Grain and Category within each Grain was tested by ANOVA (using Minitab V 17.1.0). This treats the integer number of alkaloids (0 to 12) as continuous, an approximation justified by well behaved residuals (not shown). Results are shown in

Table 6; there was statistically significant evidence that the number of alkaloids depended on both Grain (p=0.002) and Category within Grain (p<0.001).

Table 6: ANOVA on number of different alkaloids present

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Grain	3	134.6	44.879	5.10	0.002
Category(Grain)	8	1044.9	130.614	14.84	0.000
Error	196	1725.5	8.803		
Total	207	2860.0			

Grouping Information Using the Tukey Method and 95% Confidence

Means that do not share a letter are significantly different.

Grain	N	Mean	Grouping
oats	10	8.12500	A
barley	53	4.33766	A B
rye	48	2.74074	B
wheat	97	2.48635	B

Category(Grain)	N	Mean	Grouping
A(barley)	28	0.2143	E
B(barley)	14	7.0714	A
C(barley)	11	5.7273	A B C
A(oats)	8	2.3750	B C D E
B(oats)	1	12.0000	A B C
C(oats)	1	10.0000	A B C D E
A(rye)	36	2.7222	B D
B(rye)	6	4.0000	A B C D E
C(rye)	6	1.5000	B C D E
A(wheat)	75	0.8400	D E
B(wheat)	21	5.6190	A C
C(wheat)	1	1.0000	A B C D E

The grouping information in

Table 6 makes clear that the evidence for between-Category differences arose principally from barley and wheat, in which category A had fewer distinct alkaloids than at least one of the other two groups. There was no statistically significant evidence for differences between Categories B and C, or between any Categories of oats and rye. This may be attributable to the small number of samples for those groups.

Semi-quantitative: ranks of values

Quantitative data analysis is hampered by the large number of "< 1" values. In such cases one approach is to use the ranks of the values, with "<1" ranked below any other value.

Table 7 shows substantial correlations between the measurands suggesting that relatively little information would be lost by looking at total alkaloid concentration rather than the 12 individual alkaloids.

Table 7: Spearmans rank correlation coefficients between measurands:
all correlation coefficients are significant at $p < 0.001$

	ergocornine	ergocorninine	ergocristine	ergocristinine
ergocorninine	0.882			
ergocristine	0.593	0.585		
ergocristinine	0.629	0.645	0.868	
ergocryptine	0.812	0.766	0.628	0.651
ergocryptinine	0.737	0.817	0.562	0.608
ergometrine	0.512	0.562	0.523	0.582
ergometrinine	0.394	0.434	0.393	0.442
ergosine	0.697	0.669	0.708	0.666
ergosinine	0.733	0.725	0.664	0.708
ergotamine	0.564	0.541	0.761	0.740
ergotaminine	0.542	0.617	0.679	0.728

	ergocryptine	ergocryptinine	ergometrine	ergometrinine
ergocryptinine	0.836			
ergometrine	0.501	0.490		
ergometrinine	0.394	0.436	0.735	
ergosine	0.719	0.615	0.507	0.393
ergosinine	0.701	0.662	0.510	0.439
ergotamine	0.662	0.609	0.463	0.350
ergotaminine	0.701	0.738	0.561	0.401

	ergosine	ergosinine	ergotamine
ergosinine	0.784		
ergotamine	0.712	0.706	
ergotaminine	0.661	0.645	0.765

The total alkaloid concentration was calculated as the sum of the 12 individual alkaloids, treating "<1" as zero. The range of total values was very wide and very skew militating against the usual parametric analysis. To test for evidence of difference in concentration without regard to the magnitude of those differences, ANOVA was performed on the rank of Total against Grain and Category within each Grain. The residuals from this ANOVA (not shown) were well distributed; the results are summarised in Table 8.

Table 8: ANOVA on ranks of Total alkaloid

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Grain	3	10363	3454	1.96	0.122
Category(Grain)	8	244269	30534	17.29	0.000
Error	196	346040	1766		
Total	207	616359			

Grouping Information Using the Tukey Method and 95% Confidence

Means that do not share a letter are significantly different.

Category(Grain)	N	Mean Rank	Grouping
A(barley)	28	65.768	D
B(barley)	14	169.321	A
C(barley)	11	160.455	A B
A(oats)	8	100.437	B C D
B(oats)	1	205.000	A B C D
C(oats)	1	189.000	A B C D
A(rye)	36	115.069	B C
B(rye)	6	155.083	A B C
C(rye)	6	83.500	C D
A(wheat)	75	76.967	D
B(wheat)	21	147.333	A B C
C(wheat)	1	121.000	A B C D

There was no statistically significant evidence (p=0.122) of difference in Total alkaloid between Grains. There was strong evidence (p<0.001) of differences between Categories

within Grain. The grouping showed that that evidence came from barley and wheat in which Category A had lower Total alkaloid than at least one of the other two groups. There was no statistically significant evidence for differences between Categories B and C, or between any Categories of oats and rye. This may be attributable to the small number of samples for those groups.

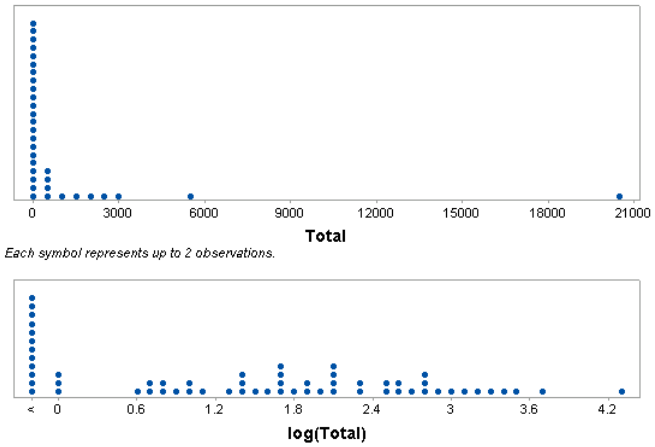
Fully quantitative

Analysis with respect to the magnitudes of values was hampered by the presence of many "<1" results. rain and Category within Grain.

Table 3 makes clear that the proportion "<1" values is much larger for Category A (1 - 42/147 = 71%) than for Categories B and C (1 - 49/61 = 20%). As the difference between Categories B and C was a principal objective fully quantitative data analysis was restricted to that subset of the data; 61 samples.

For this subset the range of total values was very wide and very skew; $\log_{10}$(Total) was distributed much more symmetrically, Figure 2. In taking logs "< 1" values were treated as $1/\sqrt{2}$ in accordance with Hornung et al.[2]. This approach is somewhat suspect with as many as 20% such values, but allows simpler analysis than alternative approaches such as maximum likelihood.

Figure 2: Distributions of Total alkaloids and $\log_{10}$(Total); Categories B and C



ANOVA of log(Total) by Grain and Category within Grain gave well behaved residuals (not shown); results are shown in Table 9.

Table 9: ANOVA on log(Total alkaloids)

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Grain	3	14.09	4.695	3.60	0.019
Category(Grain)	4	10.14	2.535	1.94	0.117
Error	53	69.13	1.304		
Total	60	91.67			

Tukey Simultaneous Tests for Differences of Means

Difference of Category(Grain)	Levels	Difference of Means	SE of Difference	Simultaneous 95% CI	Adjusted T-Value	Adjusted P-Value
C(barley) - B(barley)		-0.327	0.460	(-1.778, 1.125)	-0.71	0.996
C(oats) - B(oats)		-1.06	1.62	(-6.15, 4.04)	-0.65	0.998
C(rye) - B(rye)		-1.481	0.659	(-3.560, 0.599)	-2.25	0.343
C(wheat) - B(wheat)		-1.57	1.17	(-5.26, 2.12)	-1.34	0.878

Individual confidence level = 99.73%

[2] Richard W. Hornung and Laurence D. Reed. Estimation of Average Concentration in the Presence of Nondetectable Values. Appl.Occup.Environ.Hyg. 5 (1):46-51, 1990.

Although there was weak evidence (p=0.019) of overall differences between Grains, the evidence for differences between any two Grains failed to reach conventional statistical significance levels (P > 0.05).
There was no statistically significant evidence (p=0.117) of difference between Categories B and C within each Grain. The indications were that $\log_{10}$(Total) for Category C was lower than Category B by about one (that is, about a factor of 10 in Total). However numbers of data were small and the data was consistent with a wide range of differences, including in the reverse direction.

Conclusions

Samples were grouped into Grains and Categories as shown in
Table 10. The geometric means of the total alkaloid concentration and the numbers of samples in each group are shown in Table 11.

Table 10: Grouping of samples: text description

		Category		
		A	B	C
Grain	barley	-	rejected	rejected-cleaned
	oats	-	rejected	rejected cleaned by supplier
	rye	-	-precleaned	-cleaned
	wheat	-	rejected	rejected-cleaned

Table 11: Geometrics mean of Total alkaloid concentration:
mean (number of samples); '<1' treated as 1/√2

		Category			
		A	B	C	All
Grain	barley	1 (28)	94 (14)	44 (11)	7 (53)
	oats	4 (8)	2501 (1)	219 (1)	11 (10)
	rye	4 (36)	34 (6)	1 (6)	4 (48)
	wheat	1 (75)	37 (21)	1 (1)	3 (97)
	All	2 (147)	55 (42)	12 (19)	4 (208)

For barley and wheat there was strong evidence that alkaloid contamination was lower in Category A than in Categories B and C. For oats and rye the evidence for similar differences was not statistically significant; the indications may be artefacts of the small sample numbers.
Although the sample data suggested that Category C contamination was lower than that of Category B, the data was consistent with no difference or even differences in the other direction. More samples in those categories would be needed to reduce the uncertainty; with this number of samples only very large differences would have reached statistical significance.

References

1. Survey on ergot alkaloids in cereals intended for human consumption and animal feeding. SCIENTIFIC REPORT submitted to EFSA. Prepared by José Diana Di Mavungu, Daria A. Larionova, Svetlana V. Malysheva, Carlos Van Peteghem, Sarah De Saeger.
2. R. Krska, G. Stubbings, R. McArthur, C. Crews. A rapid LC/MS/MS method for the determination of 6 major ergot alkaloids and their epimers. Anal. Bioanal. Chem, 2008, May, 391(2), 563-76

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