



Project Report No. 652

Monitoring of mycotoxins and other contaminants in UK cereals used in malting, milling and animal feed

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This is the final report of an 84-month project which started in August 2016. The work was funded by AHDB with a grant of £813,368.

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1. GLOSSARY

AF	Aflatoxin
AHDB	Agriculture and Horticulture Development Board
AIC	The Agricultural Industries Confederation
BOBMA	The British Oat & Barley Millers Association
BSI	British Standards Institute
CEN	Comité European de Normalization
DON	Deoxynivalenol
EFSA	The European Food Safety Authority
FSA	Food Standards Agency
GC-MS	Gas Chromatography Mass Spectrometry
GC-MS/MS	Gas Chromatography Tandem Mass Spectrometry
HPLC-FLD	High performance Liquid Chromatography-Fluorescence Detection
HRMS	High Resolution Mass Spectrometric Detection
ICES-6	Six Marker or Indicator (non-dioxin-like) PCBs
ICP-MS	Inductively Coupled Plasma Mass Spectrometry
ISO	International Standards Organisation
LC-MS/MS	Liquid Chromatography Tandem Mass Spectrometry
LOD	Limit of Detection
MAGB	Maltster's Association of Great Britain
ML	Maximum Level
MRL	Maximum Residue Limit
MU	Measurement of Uncertainty
Nabim	National Association of British and Irish Millers (previous name for UKFM)
OTA	Ochratoxin A
PAHs	Polycyclic Aromatic Hydrocarbons
PCBs	Polychlorinated Biphenyls
RL	Reporting Limit
SPE	Solid Phase Extraction
UKAS	United Kingdom Accreditation Service
UKFM	UK Flour Millers
ZEN	Zearalenone

2. Abstract

This project produced independent surveillance data on the incidence and levels of key contaminants for representative commercial samples of harvested and stored grain. The occurrence of contaminants in post-intake wheat, barley and oats and their co-products was assessed using accredited analytical methods. The project tested samples, used by milling, malting and animal feed industries, harvested from 2016 (September) to stored samples from 2023 (March). Core target contaminants and the sampling numbers were agreed by the project steering group, comprising AHDB and key trade associations: UKFM, MAGB, AIC and BOBMA. Fera Science Ltd also conducted horizon scanning of publications, including consideration of impending legislative changes and advice from specialist scientists. The core set of contaminants included pesticides (fungicides, insecticides, plant growth regulators, chlorpropham, glyphosate, and piperonyl butoxide) and mycotoxins (fusarium toxins, ochratoxin A and ergot alkaloids). In some years, additional contaminants were selected (as required): heavy metals, inorganic arsenic, alternaria toxins, sterigmatocystin, dioxins and polychlorinated biphenyls, polycyclic aromatic hydrocarbons, acrylamide, beauvericin, enniatins and aflatoxins. In general, most cereals used by UK processors adhere to both EU and UK laws and guidelines concerning the presence of the contaminants monitored in this project. The main findings were:

- DON was the most frequently detected trichothecene mycotoxin. It tended to be higher in oatfeed and in wheat and its co-products compared to the other commodities. Mean and maximum levels for all commodities were below the maximum limits (MLs)
- Mean results for T-2 and HT-2 mycotoxins were significantly higher in oats and oat products compared other commodities
- ZEN levels tended to be low, with the only ML exceedances found in two samples of milling wheat 2017 and 2021 and one sample of food oats in 2020
- Incidence levels were generally high for ergot alkaloids in most products (> 50%) each year. However, the actual concentrations found were generally low
- Ochratoxin A was less frequently detected in food grains, and at very low concentrations. Only four samples exceeded MLs (three food oats and one milling wheat). Wheatfeed and oatfeed concentrations were high but were still well below guideline levels
- Plant growth regulators were the most detected agri-chem residues each year. Glyphosate and some fungicides were commonly detected and showed year-on-year variance
- Insecticides were commonly detected and, over the last two years of the survey, had a noticeable decrease in their average residues per sample
- Piperonyl butoxide was commonly found and shows little variance in its detection
- Chlorpropham incidence was low but was consistently detected each year with no characteristic pattern

- Concentrations of heavy metals were generally low and well below current legal limits. Nickel monitoring data had been requested by EFSA. Although incidence was high, the concentrations measured were low

3. Introduction

This project provided an independent programme of gathering surveillance data on the incidence and levels of key contaminants (mycotoxins and others) for representative UK-grown samples of freshly harvested grain and in stored grain of the main cereals within the UK grain supply chain.

The surveillance data has been available to be used to:

- a) Inform and alert the cereal industry on the safety of their products
- b) Inform discussions on impending revision of legislation and risk assessments
- c) Demonstrate due diligence compliance
- d) Provide scientific evidence to support the assertion that UK cereal products are safe and wholesome both to the domestic and export markets.

The project was set up initially for a five-year period but has been extended three times for one year each time. This report summarises the results of Years 1 – 7. Annual reports of each year's results have already been published on the AHDB website (<https://ahdb.org.uk/monitoring-of-contaminants-in-uk-cereals-used-for-processing-food-and-animal-feed>).

Each year, samples of freshly harvested samples were delivered annually to the laboratory for analysis of “core” analytes, namely trichothecene mycotoxins, zearalenone, ergot alkaloids, fungicides, glyphosate and Plant Growth Regulators. Samples were also collected from stores. Samples of malt and malting barley pairs were analysed for trichothecenes, zearalenone, ochratoxin A and pesticides. The methods are fully validated and accredited by UKAS to ISO 17025.

Additional analyses of selected contaminants (other mycotoxins, metals, processing contaminants and dioxins) were carried out as requested by the project steering group, again using validated and accredited methods. In the event that any analytical results exceeded legislative or guidance values, the results were drawn to the attention of relevant personnel in the cereal industry and AHDB, the analysis repeated, and the result confirmed within one week. All samples were archived and held in secure controlled temperature rooms for up to 5 years initially but was reduced to 3 years from 2022.

An additional part of the project was the intelligence gathering on emerging issues concerning legislation, cereal contaminants and safety which may have an impact on UK grown cereals. Alerts from HorizonScan were sent to partners weekly, and a more comprehensive report was circulated every quarter highlighting upcoming changes in legislation, events and relevant scientific publications, such as new EFSA Opinions or peer reviewed papers.

Project data on contaminants (not pesticide residues) was collated and formatted into the correct format for submission to EFSA and FSA calls for analytical data.

4. Materials and methods

4.1. Sampling

A sampling plan was developed by the project committee comprising AHDB, MAGB, AIC, UKFM and BOBMA. A 'core' testing schedule was produced and agreed. The committee met prior to the harvest period each year to discuss any changes, additional testing requirements or any emerging issues which would arise throughout the year.

Typically, 2kg of post-intake samples of 9 different commodities were sent to Fera Science Ltd for analysis. There were two main collections, one in September (immediately after harvest) and one in March (6 months after storage). There were also two additional collections: one during the period of November to March and one in January. Full details of the commodities and their collection times are shown in Table 1.

Table 1. Typical annual sampling time points for freshly harvested and stored samples

Sampling Month				
	September	November - February	January	March
Milling Wheat	50		25	25
Malting Barley	40	20		
Malt		20		
Food Oats	29			30
Food Barley	1			
Feed Wheat	14			40
Wheatfeed	20			12
Feed Barley	14			36
Feed Oats	6			6
Oatfeed	6			6

Throughout the project, five core tests were carried out on all commodities received in any given year. Those were trichothecenes, zearalenone, ergot alkaloids, ochratoxin A and pesticides (including chlorpropham). The only exception was that ergot alkaloid analysis was not carried out on malt samples. Additional tests were carried out at various stages of the project. Full details are shown in Tables 2 and 3.

Table 2. Core tests carried out on samples each year

Years 1 - 7	
Commodity	Test
Milling Wheat	Trichothecenes Zearalenone Ergot Alkaloids Ochratoxin A Pesticides (Including Chlorpropham)
Malting Barley	
Malt*	
Food Oats	
Food Barley	
Feed Wheat	
Wheatfeed	
Feed Barley	
Feed Oats	
Oatfeed	

*Ergot alkaloid analysis was not carried out on malt samples.

Table 3. Additional tests carried out on samples, by commodity and year

	Year 1	Year 2	Year 3	Year 4	Year 5	Year 6	Year 7
Commodity	Additional Tests						
Milling Wheat	Metals		Metals	Beauvericin Enniatins	Metals	Metals Aflatoxins	Metals
Malting Barley	Metals Alternaria toxins					Metals Aflatoxins Chlorate & perchlorate	Metals
Malt	Metals		Acrylamide			Aflatoxins Chlorate & perchlorate	Acrylamide
Food Oats	Metals Alternaria toxins Sterigmatocystin	Metals				Metals	Metals
Food Barley	-						Metals
Feed Wheat	Metals Dioxins & PAHs	PAHs					Metals
Wheatfeed	Metals						Metals
Feed Barley	Metals Dioxins & PAHs	PAHs					Metals
Feed Oats	Metals + Inorganic Arsenic Dioxins & PAHs	Metals PAHs					Metals
Oatfeed							Metals

4.2. Methods

4.2.1. Mycotoxins analysis

Multi-Mycotoxin analysis by LC-MS/MS

The in-house method FSG 818 “Method for the extraction and LC-MS/MS analysis of 17 mycotoxins (AHDB suite)” was used to analyse the following mycotoxins (with respective reporting limits in µg/kg shown in brackets): 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), T-2 toxin- α 3-glucoside (10), α -zearalenol (2.5), β -zearalenol (2.5), α -zearalenol-14-glucoside (5), β -zearalenol-14-glucoside (5), zearalenone (2.5), and zearalenone-14-glucoside (5).

Samples were extracted with a mixture of acetonitrile and water, cleaned-up by solid phase extraction (SPE), resuspended in a mix of 7 isotopically-labelled mycotoxins (for use as internal standards) in acetonitrile and water, then analysed by LC-MS/MS. A blank sample and spiked samples (and an in-house reference sample where available) were included in the batches as quality controls.

Ochratoxin A analysis by HPLC-FLD

Analysis of ochratoxin A was carried out using in-house SOP FSG 252 “Determination of Ochratoxin A using immunoaffinity column clean-up and HPLC”. The reporting limit is 0.2 µg/kg, the analysis is accredited to ISO 17025 [1].

Samples were extracted with a mixture of acetonitrile and water, cleaned-up by immunoaffinity column and analysed using reverse phase HPLC, with fluorescence detection. A blank sample, two spiked samples, and an in-house reference sample were included in the batches as quality controls and to determine recoveries.

Ergot Alkaloid analysis by LC-MS/MS

Analyses of six ergot alkaloids (ergometrine, ergosine, ergocornine, ergocryptine, ergotamine, ergocristine) and their -inine epimers were carried out using in-house SOP FSG 601 “Determination of Ergot Alkaloids in Cereals and Cereal Products by LC-MS/MS”. The reporting limit for each analyte is 0.5 µg/kg, and the analysis is accredited to ISO 17025 [2].

Ergot alkaloids were extracted into an organic solvent and the extracts cleaned up using bonded phase SPE material. The extracts were analysed by HPLC with tandem mass spectrometry and levels compared with authentic standards. A blank sample, two spiked samples, and an in-house reference sample were included in the batches as quality controls and to determine recoveries.

Mycotoxin analyses by HPLC-FLD

Analysis for aflatoxins B₁, B₂, G₁ and G₂ and ochratoxin A was carried out using in-house SOP FSG 261 Simultaneous determination of ochratoxin A and aflatoxins B₁, B₂, G₁ and G₂ using immunoaffinity column clean-up and HPLC with fluorescence detection (HPLC-FLD) [3]. The reporting limit for each analyte is 0.2 µg/kg, the analysis is accredited to ISO 17025.

Samples were extracted with a mixture of acetonitrile and water, cleaned-up by immunoaffinity column and analysed using reverse phase HPLC, with a gradient elution and fluorescence detector programmed to detect aflatoxins and ochratoxin A. A blank sample and two spiked samples (in the absence of an in-house reference sample) were included in the batches as quality control samples.

Alternaria Toxins analysis by LC-MS/MS

Five *Alternaria* toxins were analysed using the CEN method EN 17521:2021 (from the method validation study of Gonçalves et al., 2022, BSI, 2021) [4, 5]. Reporting limits are 1 µg/kg for alternuene, alternariol, and alternariol monomethyl ether; 5 µg/kg for tentoxin; and 10 µg/kg for tenuazonic acid.

A mixture of methanol, water, and acetic acid was used for extraction, with a polymeric based solid-phase extraction cartridge used for clean-up. The extracts were then analysed by HPLC with tandem mass spectrometry (LC-MS/MS). A blank sample and two spiked samples (in the absence of an in-house reference sample) were included in the batches as quality controls and to determine recoveries.

Sterigmatocystin analysis by LC-MS/MS

Analysis of sterigmatocystin was carried out using an in-house SOP "Analysis of Sterigmatocystin using LC-MSMS". The reporting limit is 0.2 µg/kg.

The analyte was extracted with a mixture of acetonitrile and water, with ¹³C-labelled sterigmatocystin added as an internal standard. The extracts were analysed by HPLC with tandem mass spectrometry (LC-MS/MS). A blank sample, two spiked samples, and an in-house reference sample were included in the batches as quality controls.

Beauvericin and Enniatins

In 2019 analysis of beauvericin and enniatins was carried out using an in-house SOP "Analysis of Beauvericin and Enniatins using LC-MSMS". Beauvericin and Enniatins A, A1, B, and B1 were analysed with reporting limits of 1 µg/kg each.

The analytes were extracted with a mixture of acetonitrile : water : acetic acid. The extracts were analysed by LC-MS/MS. A blank sample and two spiked samples (in the absence of an in-house reference sample) were included in the batches as quality controls and to determine recoveries.

Acrylamide analysis by GC-MS

Analysis of acrylamide was carried out using in-house SOP 262 “Determination of Acrylamide in foods and drinks by GC-MS” based on BS PD CEN/TS 17083:2017 (BSI, 2017) [6]. For malt, the reporting limit was 30 µg/kg.

Samples were extracted with hot water. The aqueous extract was brominated, solvent extracted, concentrated, then analysed by gas chromatography with mass spectrometry detection (GC-MS). ¹³C-acrylamide was used as an internal standard, which gives an implicit correction for recovery. A blank sample, two spiked samples, and an in-house reference sample were included in the batches as quality controls.

4.2.2. Pesticide analysis

Samples were analysed for over 400 pesticides using two in-house multi-residue screening methods. For in-house method FSG/167 - LCMS - a sub-sample was extracted with acetonitrile, in the presence of salts. Analysis was carried out using liquid chromatography with mass spectrometric detection (LC-MS/MS) in selected reaction monitoring mode. The presence of residues was confirmed using the same technique in multiple reaction monitoring mode.

For in-house method FSG/167 - GCMS – a sub-sample was extracted with acetonitrile, in the presence of salts. After clean-up using dispersive SPE, analysis was carried out using gas chromatography with mass spectrometric detection (GC-MS/MS) in selected reaction monitoring mode.

A full list of the analytes included in the methods and their reporting limits is given in Appendix 1.

4.2.3. Metals analysis (including Inorganic Arsenic)

Metals analyses

Samples were analysed for aluminium, nickel, copper, arsenic, cadmium, mercury and lead by following in-house methods; FSG 461, FSG 457 and FSG 463. Aliquots of homogenised test sample were digested in a mixture of concentrated nitric acid and hydrochloric acid using a high-pressure microwave system. Quantification was by inductively coupled plasma-mass spectrometry (ICP-MS) with collision cell. Quality checks included blanks, spikes and certified reference materials.

All data were corrected for reagent blank and spike recovery. The Reporting Limit was equal to, or exceeded, 10 x standard deviation of reagent blank values adjusted for dilution and sample weight. Reference material results were all satisfactory. Results are UKAS accredited (ISO 17025).

Metals analysis – Inorganic Arsenic

Samples were analysed for Inorganic Arsenic using in-house method FSG 456. Aliquots of homogenised test sample were solubilised using concentrated hydrochloric acid at room temperature whereby the inorganic arsenic species are converted to covalent halides. As(V) species were reduced to As(III) using hydrobromic acid and hydrazine sulphate and the covalent halide was then extracted into chloroform. The arsenic was back extracted from the chloroform into dilute hydrochloric acid. Quantification was by inductively coupled plasma-mass spectrometry (ICP-MS) with collision cell. Quality checks included blanks, spikes and certified reference materials.

4.2.4. Dioxins and PAH Analysis

Dioxins, PCBs, Polychlorinated dibenzofurans (PCDFs) analyses by HRMS

In-house methods FSG 403 to 408 were used for the detection of Dioxins and PCBs, the methods are applicable for animal feeds, beverages, foods and human tissues, crops, soils, pollutants and effluents (solid). The methods are UKAS accredited to the ISO 17025 Standard.

An aliquot of each sample was fortified with known amounts of surrogate (¹³C₁₂-labelled) analogues of target analytes and exhaustively extracted using mixed organic solvents. The extract was cleaned up using adsorption chromatography. Ortho-PCBs, non-ortho-PCBs and PCDDs/PCDFs were segregated into two separate fractions. Each fraction was then concentrated and further cleaned-up before the inclusion of additional surrogate standards. Final determination was conducted by high resolution gas chromatography with either low resolution mass spectrometric detection (ortho-PCBs) or high-resolution mass spectrometric detection (HRMS) (non-ortho-PCBs and PCDDs/PCDFs).

Polycyclic aromatic hydrocarbons (PAHs) analyses

Samples were analysed for Polycyclic Aromatic Hydrocarbons (PAHs) using in-house method FSG 410 Extraction of Foods for the Determination of PAHs, accredited to ISO 17025. The method can determine 28 PAHs, including the four regulated (marker) PAH compounds benzo[a]pyrene (BAP), benzo[a]anthracene, benzo[b]fluoranthene and chrysene. The full list of analytes included in the method is:

- acenaphthylene
- acenaphthene
- fluorene
- anthracene

- phenanthrene
- fluoranthene
- benzo[c]fluorene
- pyrene
- **benzo[a]anthracene***
- benzo[ghi]fluoranthene
- benzo[b]naphtho[2,1-d]thiophene
- cyclopenta[c,d]pyrene
- **chrysene***
- 5-methylchrysene
- **benzo-[b]-fluoranthene***
- benzo[j]fluoranthene
- benzo[k]fluoranthene
- benzo[e]pyrene
- **benzo[a]pyrene***
- indeno[1,2,3-cd] pyrene
- dibenz[ah]anthracene
- benzo[g,h,i]perylene
- anthanthrene
- dibenzo[a,l]pyrene
- dibenzo[a,e]pyrene
- dibenzo[a,i]pyrene
- dibenzo[a,h]pyrene
- coronene

*These four regulated PAHs are included in the PAH4 SUM, in Retained Regulation (EC) 1881/2006 [7].

An aliquot of the homogenised sample was fortified with appropriate ¹³C Internal standards and subjected to saponification followed by liquid-liquid extraction. Clean up was by dimethylformamide/cyclohexane partition followed by elution through a silica gel column. Analysis was by gas chromatography mass spectrometry (GC-MS).

5. Measurement of uncertainty

Scientific uncertainty is a quantitative measurement of variability in the data. In other words, uncertainty in science refers to the idea that all data have a range of expected values as opposed to a precise point value.

For all analysis carried out throughout the project, measure of uncertainty (MU) was available, if requested, in-line with requirements set out in ISO17025, for which the methods are accredited. MU is updated regularly as more recent data becomes available. Therefore, the MU's listed below are correct for at the time of writing. MU are provided for the 'core' analytes.

Method	Measurement of Uncertainty (%)
Trichothecenes	35-50
Zearalenone	50
Ergot Alkaloids	55
Ochratoxin A	50
Pesticides	50

6. Results

Full results from every year of the project are available from the individual annual reports at the project website:

<https://ahdb.org.uk/monitoring-of-contaminants-in-uk-cereals-used-for-processing-food-and-animal-feed>

6.1. Deoxynivalenol

6.1.1. Harvest

Incidence, mean, maximum and median values for all commodities over the full 7 years are displayed in tables 4 and 5 and Figure 1.

When comparing mean values for DON throughout this project, there was no obvious pattern or trend for almost all commodities. It is also difficult to draw conclusions for some commodities as the sample set size was so small, e.g. for food barley, only one sample per year was submitted. In most years, feed products were responsible for the maximum DON level found, and wheatfeed consistently had the highest mean DON levels. However, in both 2016 and 2019, milling wheat contained the highest maximum concentration. None of the feed samples exceeded the guidance level for feed of 8 mg/kg DON in cereals and cereal products in EU Recommendation 576/2006 [8]. In 2017, a sample of milling wheat contained 1540 µg/kg DON (mean result, n=3) this did not exceed the ML of 1250 µg/kg in (Assimilated) Regulation (EC) No 1881/2006 [7] when measurement uncertainty (MU) was taken into account.

For several commodities, the lowest mean and maximum values were observed in 2022 or were similar to the lowest mean and maximum levels observed in other years.

Mean and maximum levels for all commodities were below the ML throughout the 7 years.

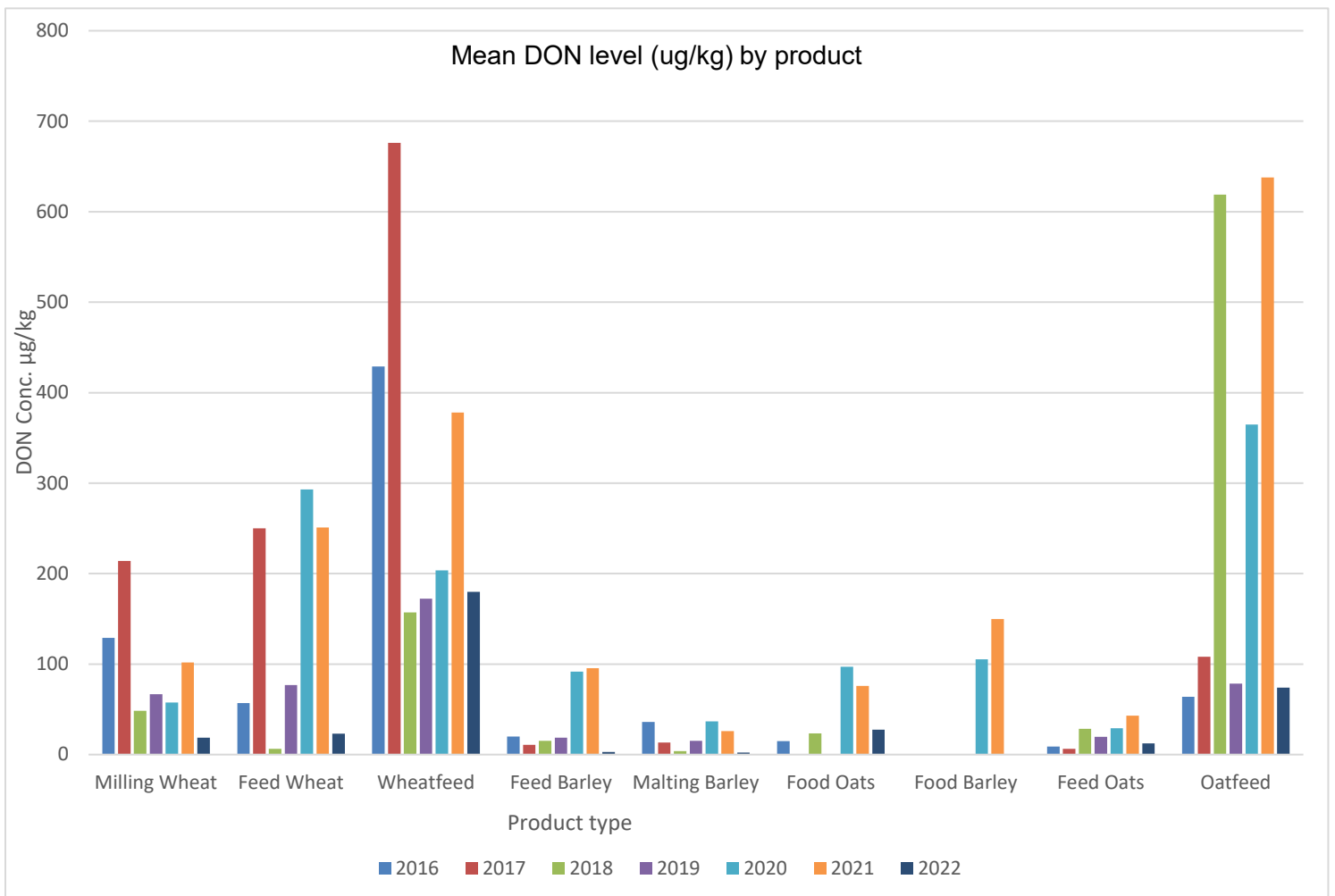
Table 4. Summarised results for DON for 2016–2018

	2016				2017				2018			
	>RL	Mean	Max	Med	>RL	Mean	Max	Med	>RL	Mean	Max	Med
	%	µg/kg			%	µg/kg			%	µg/kg		
Milling Wheat	96	129	1006	54	98	214	1540	108	50	49	420	5
Feed Wheat	80	57	180	48	100	250	1127	171	9	6	70	<10
Wheatfeed	100	429	819	478	100	676	2016	426	100	157	502	124
Feed Barley	33	20	85	<10	36	11	59	<10	60	15	45	14
Malting Barley	60	36	117	29	48	13	109	<10	20	4	40	<10
Food Oats	23	15	132	<10	7	1	12	<10	66	23	160	18
Food Barley	n/a	n/a	n/a	n/a	0	n/a	<10	n/a	n/a	n/a	n/a	n/a
Feed Oats	40	9	33	<10	27	7	39	<10	46	29	231	<10
Oatfeed	100	64	332	37	100	108	611	49	100	619	2158	261

Table 5. Summarised results for DON for 2019–2022

	2019				2020				2021				2022			
	>RL	Mean	Max	Med	>RL	Mean	Max	Med	>RL	Mean	Max	Med	>RL	Mean	Max	Med
	%	µg/kg			%	µg/kg			%	µg/kg			%	µg/kg		
Milling Wheat	76	67	798	25	88	58	537	27	90	102	620	61	54	19	174	11
Feed Wheat	90	77	301	51	90	293	1575	24	80	251	1414	95	43	23	127	<10
Wheatfeed	100	172	459	133	100	204	676	180	100	378	1485	289	100	180	546	141
Feed Barley	70	19	100	12	80	92	421	50	67	96	790	30	14	3	27	<10
Malting Barley	60	15	77	14	69	37	176	19	60	26	201	12	10	2	39	<10
Food Oats	17	<10	72	<10	45	97	1535	<10	55	76	746	12	52	28	134	12
Food Barley	0	<10	<10	<10	100	105	105	105	100	150	150	150	0	<10	<10	<10
Feed Oats	40	20	102	<10	80	29	73	25	100	43	120	28	50	13	31	6
Oatfeed	100	79	213	66	100	365	776	418	100	638	2581	44	83	74	133	88

Figure 1. Mean DON concentrations (µg/kg) by product type



6.1.2. Stored

As well as freshly harvested malting barley, matched pairs of malting barley from store and the malt produced from it were analysed each year for trichothecenes and zearalenone.

These samples in general produced a lot of results below the RL (10 µg/kg). Mean and maximum values for DON tended to be higher in malting barley at the beginning of the project (2016/2017); however, the highest concentration and highest frequency of residues were found in the samples in Year 5 (2020-21). Summarising the results in Year 1 (2016-17), 12/20 malting barleys were above the RL, 5/20 malt samples contained residues, the maximum level found was 88 µg/kg.

In Year 2 (2017-18), 9/20 malting barleys contained DON, the maximum level was 178 µg/kg, and 5 corresponding malts contained DON. There was no consistent pattern, 2 malts contained higher levels of DON (both 3 times barley level), 2 were lower (about half the barley level), one was the same. The other four were below the RL.

In Year 3 (2019-20), virtually no DON was detected, with only 3 malting barley samples just above the RL and one malt at 41.4 µg/kg.

Year 4 (2020-21), 11/20 barley samples and their corresponding malts contained DON. No patterns were observed, in some cases the DON levels in the malts were higher, lower and the same as the barley they were produced from. Also, 3 malting barley samples contained DON, but the malt made from those was <RL, while 2 malt samples contained DON but the barley pair was <RL. The maximum level found was 237 µg/kg in malt, the matching barley contained 184 µg/kg.

In Year 5 (2021-22), 5 malting barleys were above RL, the maximum level was 47.6 µg/kg, 1 malt just above RL – its matching barley was <RL.

For Year 6 (2022-23), 9 malting barley samples contained DON (maximum level 133 µg/kg), 6 malts contained DON above RL, the maximum level of 111 µg/kg was found in malt prepared from barley that contained 110 µg/kg.

6.2. T-2 & HT-2 toxins

6.2.1. Harvest

Incidence, mean, maximum and median values for all commodities over the 7 years are displayed in tables 6 and 7. Mean results for T-2 and HT-2 toxins were significantly higher in oats and oat products. Figures 2 and 3 display the mean results over the 7 years of the project with the products separated according to the concentrations found, Figure 2 presents results for oats products and Figure 3 presents the results for the other products.

Generally, the mean values for all commodities were relatively consistent throughout the life of the project and there appears to be no obvious increasing or decreasing trends, apart from oatfeed, where the mean value has generally decreased since 2018, with one unusually high year (2021). There were no MLs for T-2 and HT-2 toxins up to 2022, although Indicative levels were set by Commission Recommendation 2013/165/EU [9], setting levels at 1000 µg/kg for oats (with husk) 100 µg/kg (wheat), 200 µg/kg (barley including malting barley) and 2000 µg/kg for Oat milling products (husks) for feed.

Throughout the project, there have been several occasions where samples exceeded these levels. The Food Standards Agency called for data on T-2 and HT-2 toxin in 2023. All T-2 and HT-2 toxin results from the project has been submitted. This data will be used by the FSA to carry out their own risk assessment on T-2 and HT-2 toxins.

Table 6. Summarised results for T-2 and HT-2 toxins in year 2016–18

	2016				2017				2018			
	>RL	Mean	Max	Med	>RL	Mean	Max	Med	>RL	Mean	Max	Med
	%	µg/kg			%	µg/kg			%	µg/kg		
Milling Wheat	0	<20	<20	<20	8	2.9	64	<20	10	4.7	139	<20
Feed Wheat	20	5	32	<20	0	<20	<20	<20	0	<20	<20	<20
Wheatfeed	0	<40	<40	<40	32	8.9	52	<20	33	7.9	30.7	<20
Feed Barley	0	<40	<40	<40	27	18.2	70	<20	40	40.3	260	<20
Malting Barley	38	10	91	<10	3	0.8	30	<20	65	37.6	210	<20
Food Oats	70	173	1093	77	97	478	1837	278	100	443	2745	188
Food Barley	n/a	n/a	n/a	n/a	0	<20	<20	n/a	n/a	n/a	n/a	n/a
Feed Oats	100	115	437	65	82	225	716	82	92	114	582	49
Oatfeed	100	1761	5787	1366	100	1038	2091	981	100	1299	4192	676

Table 7. Summarised results for T-2 and HT-2 toxins in year 2019–22

	2019				2020				2021				2022			
	>RL	Mean	Max	Med	>RL	Mean	Max	Med	>RL	Mean	Max	Med	>RL	Mean	Max	Med
	%	µg/kg			%	µg/kg			%	µg/kg			%	µg/kg		
Milling Wheat	6	<20	43	<20	4	<20	50.2	<20	4	1.2	43	<20	2	0.2	10	<20
Feed Wheat	0	<20	<20	<20	0	<20	<20	<20	0	<20	<20	<20	0	<20	<20	<20
Wheatfeed	20	9.1	86	<20	35	10.5	58.5	<20	83	15.8	38	16	50	10	38	5
Feed Barley	20	9.4	62.6	<20	10	2.1	21	<20	53	24.9	143	<20	0	<20	<20	<20
Malting Barley	45	12.1	63	<20	6	6	190	<20	50	19.6	302	<20	23	<20	129	<20
Food Oats	93	458	2391	224	86	313	1355	106	100	357	1030	264	83	433	3283	87
Food Barley	0	<20	<20	<20	0	<20	<20	<20	100	30.5	31	31	100	23	23	23
Feed Oats	80	246	2077	37	60	183	466	124	100	213	337	262	100	252	615	181
Oatfeed	100	1237	2143	1132	100	1132	1956	1092	100	1722	4734	937	100	770	1095	751

Figure 2. Mean Sum T-2 and HT-2 toxin concentration (µg/kg) in oat products

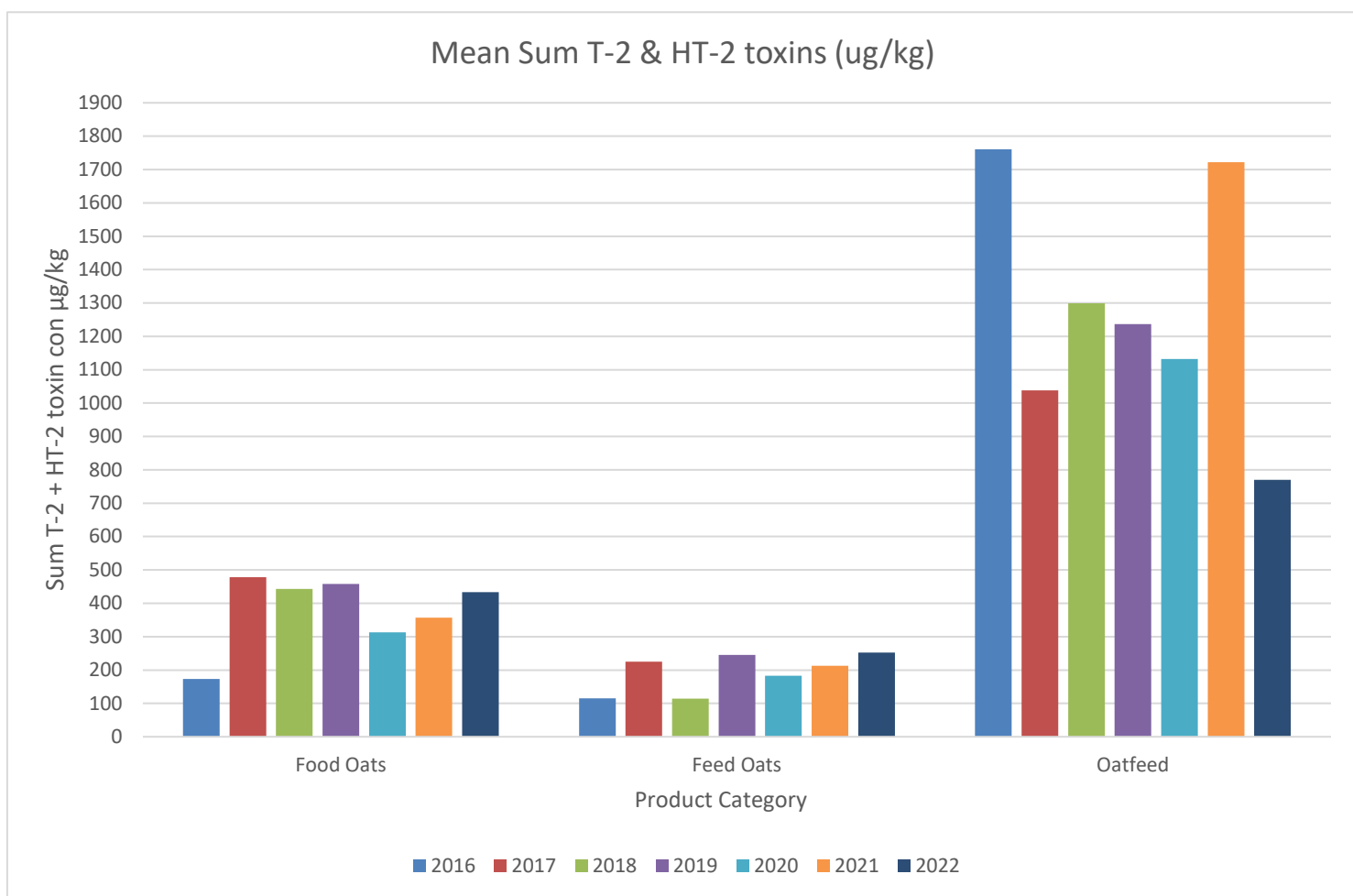
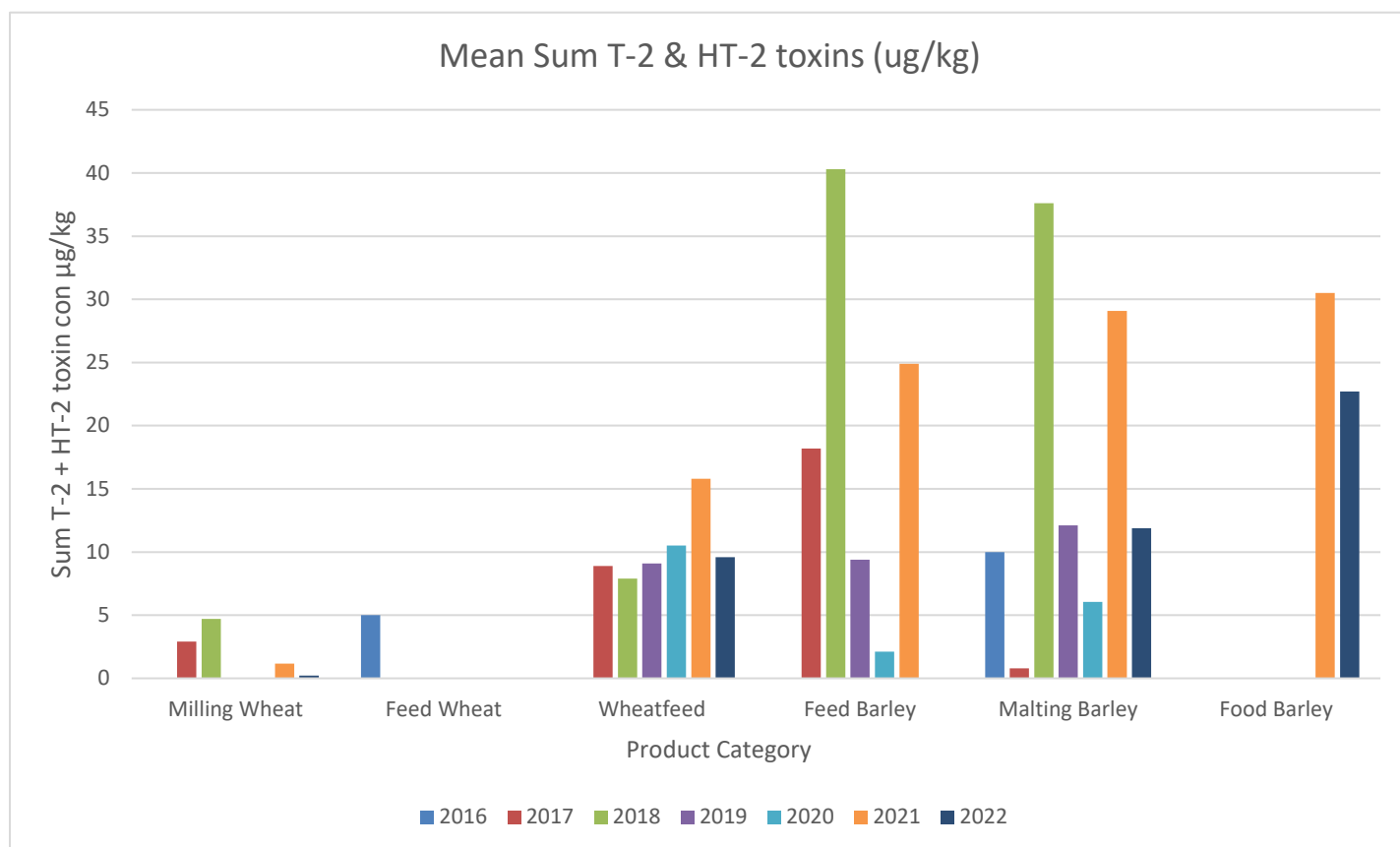


Figure 3. Mean Sum T-2 and HT-2 toxin concentration (µg/kg) in other cereal products



6.2.2. Stored

Stored malting barley, along with a sample of malt which was produced from each barley sample were analysed each year. Very little was observed throughout the project, with only one sample exceeding the guidance levels for T-2 and HT-2 in malting barley (282 µg/kg).

6.3. Zearalenone

6.3.1. Harvest

Incidence, mean, maximum and median values for all commodities over the 7 years of the project are displayed in tables 8 and 9 with mean ZEN levels displayed in Figure 4.

Although incidence levels of ZEN generally increased throughout the project (peaking in 2020/2021), mean and median levels have remained low. There have only been two instances of mean ZEN above 40 µg/kg throughout the project, once in 2017 (114 µg/kg - feed wheat) and once in 2018 (71.8 µg/kg - oatfeed). On both occasions, the median values were much lower (29.1 µg/kg and 11.3 µg/kg, respectively).

ML exceedances have been very few for ZEN, with only 3 confirmed results throughout the 7 years of testing. These were two samples of milling wheat with values of 327 µg/kg (2017) and 119 µg/kg (2021) and 1 sample of food oats with a value of 948 µg/kg (2020). The sample from 2017 was the sample that also contained 1540 µg/kg DON. This was UK-grown Crusoe wheat, and no information on the area where it was grown was provided. The milling wheat from 2021 was grown in England, (location not provided).

6.3.2. Stored

A second set of malting barley were analysed each year, along with a sample of malt which was produced from each barley sample. Very few residues were detected in any of the samples and the levels measured were all low. For example, in Year 1 a sample of malting barley contained 6 µg/kg. In Year 2, one malting barley contained 17.7 µg/kg, and one malting barley contained 23.7 µg/kg and its corresponding malt had a level of 9.6 µg/kg. ZEN was not detected in malting barley and malt in Year 3. In Year 4, 4 samples contained ZEN with a maximum level of 10.2 µg/kg. Year 5 had the most samples with residues, 5 samples of malting barley and 6 samples of malt – ZEN was in 5 matched pairs, plus an extra malt sample. The levels in the malting barley and malt pairs were mostly similar or higher in the malt. The maximum level found was 25.4 µg/kg in a malt. In Year 6, one sample of malting barley and 2 malts contained residues, but there was no correlation. The maximum level found was 9.6 µg/kg in the malting barley, and in Year 7, ZEN was not detected in any malting barley or malt.

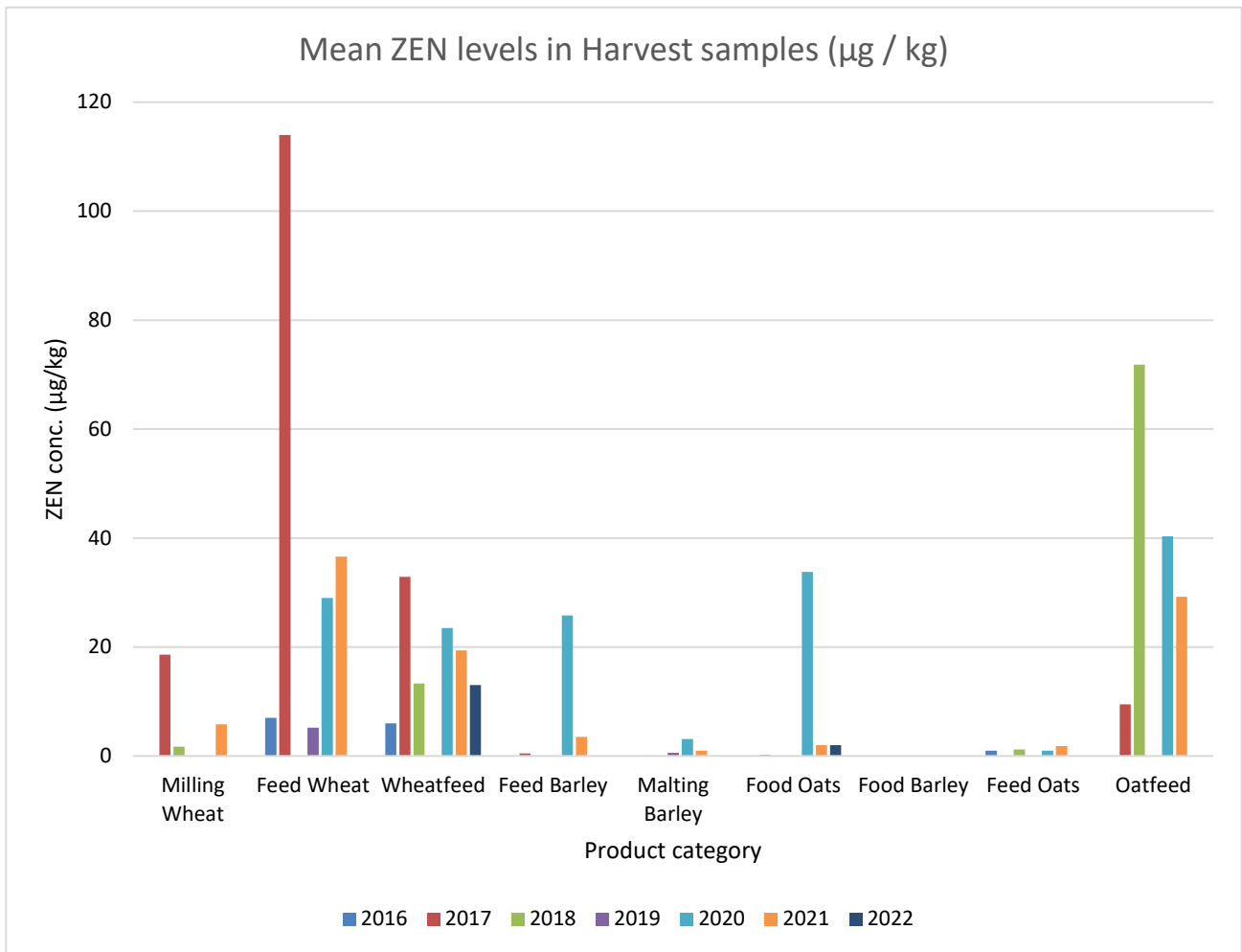
Table 8. Zearalenone results 2016–2018 in harvest samples

	2016				2017				2018			
	>RL	Mean	Max	Med	>RL	Mean	Max	Med	>RL	Mean	Max	Med
	%	µg/kg			%	µg/kg			%	µg/kg		
Milling Wheat	24	<2.5	17	<2.5	70	18.6	327	7.4	12	1.7	22	<2.5
Feed Wheat	50	7	23	4	82	114	916	29.1	0	<2.5	<2.5	<2.5
Wheatfeed	50	6	33	3	95	32.9	94.7	25.5	71	13.3	68.6	11.2
Feed Barley	0	<25	<25	<25	9	0.5	5.5	<2.5	10	<2.5	4.5	<2.5
Malting Barley	3	<2.5	6	<2.5	3	0.1	3	<2.5	0	<2.5	<2.5	<2.5
Food Oats	3	<2.5	4	<2.5	3	0.2	6	<2.5	0	<2.5	<2.5	<2.5
Food Barley	n/a	n/a	n/a	n/a	0	n/a	<2.5	n/a	n/a	n/a	n/a	n/a
Feed Oats	20	1	8	<2.5	0	<2.5	<2.5	<2.5	8	1.2	15.1	<2.5
Oatfeed	0	<25	< 25	<25	50	9.5	63.5	1.3	60	71.8	269	11.3

Table 9. Zearalenone results 2019–2022 in harvest samples

	2019				2020				2021				2022			
	>RL	Mean	Max	Med	>RL	Mean	Max	Med	>RL	Mean	Max	Med	>RL	Mean	Max	Med
	%	µg/kg			%	µg/kg			%	µg/kg			%	µg/kg		
Milling Wheat	22	<2.5	19	<2.5	16	<2.5	37.2	<2.5	35	5.8	115	<2.5	2	0.06	3.2	<2.5
Feed Wheat	70	5.2	12.9	5.5	50	29	191	1.9	53	37	353	2.7	0	<2.5	<2.5	<2.5
Wheat feed	0	<2.5	<2.5	<2.5	80	24	178	11	94	19	60	19	75	13	36	11
Feed Barley	0	<2.5	<2.5	<2.5	80	26	114	9.9	33	3.5	22	<2.5	0	<2.5	<2.5	<2.5
Malting Barley	13	0.6	9.1	<2.5	31	3.1	28	<2.5	8	1	11	<2.5	0	<2.5	<2.5	<2.5
Food Oats	3	<5	7.5	<5	24	34	948	<2.5	28	2	33	<2.5	3	2	60	<2.5
Food Barley	1	<5	<5	<5	0	<2.5	<2.5	<2.5	0	<2.5	<2.5	<2.5	0	<2.5	<2.5	<2.5
Feed Oats	0	<2.5	<2.5	<2.5	30	1	4	<2.5	33	1.8	7.4	<2.5	0	<2.5	<2.5	<2.5
Oat feed	0	<2.5	<2.5	<2.5	80	40	102	41	67	29	134	6.8	33	1	3	<2.5

Figure 4. Mean ZEN levels in harvest samples



6.4. Ergot Alkaloids

Incidence, mean, maximum and median values for all commodities over the 7 years of the project are displayed in Tables 10 and 11 with mean total ergot alkaloid levels displayed in Figure 5.

Although only the sum of total ergots is displayed in the tables and charts, it is worth noting that the method measured 12 compounds, and typically samples contained multiple ergot alkaloids and also several occasions of samples containing all six alkaloids and epimers.

Broadly speaking, incidence levels for all products were reasonably high (>50%) throughout the project. However, oatfeed and wheatfeed were consistently the most contaminated commodities with incidence levels of >83%.

Mean, maximum and median levels for total ergot alkaloids have remained consistent and low for oatfeed, feed oats, food oats and malting barley, with the highest maximum being 710 µg/kg in a 2016 food oat sample.

Milling wheat has generally had consistent mean values, hovering around 50 µg/kg, with one standout year (2021) returning a mean value of 213 µg/kg. Even though the mean value was high in 2021, the median value was <6.0, suggesting the mean value was inflated due to a small number of high values. Indeed, 4 samples of milling wheat had values >1000 µg/kg, and all other samples returned significantly lower values.

Wheatfeed and feed barley mean and maximum levels have varied quite significantly in some years. Wheatfeed mean levels have ranged from 62.3 µg/kg (2018) to 454 µg/kg (2021), with maximum values ranging from 248 µg/kg (2019) to 1119 µg/kg (2021). Feed barley mean values have ranged from <6 µg/kg (2017) to 466 µg/kg (2021) with maximum values ranging from 32.1 µg/kg (2018) to 6037 µg/kg (2021). It is important to state that all occasions, the median values for all commodities were much lower than the mean, and the high mean value of 2021 was driven by the one exceptional result.

Regulation (EU) 2021/1399 introduced maximum levels for ergot alkaloids in the EU but these levels do not apply within GB [10]. Fera did conduct duplicate analysis of results considered to be 'high', i.e. exceed the EU maximum levels for cereals for food products, however, the number of incidence of samples at these concentrations was very low (<1% of all samples received).

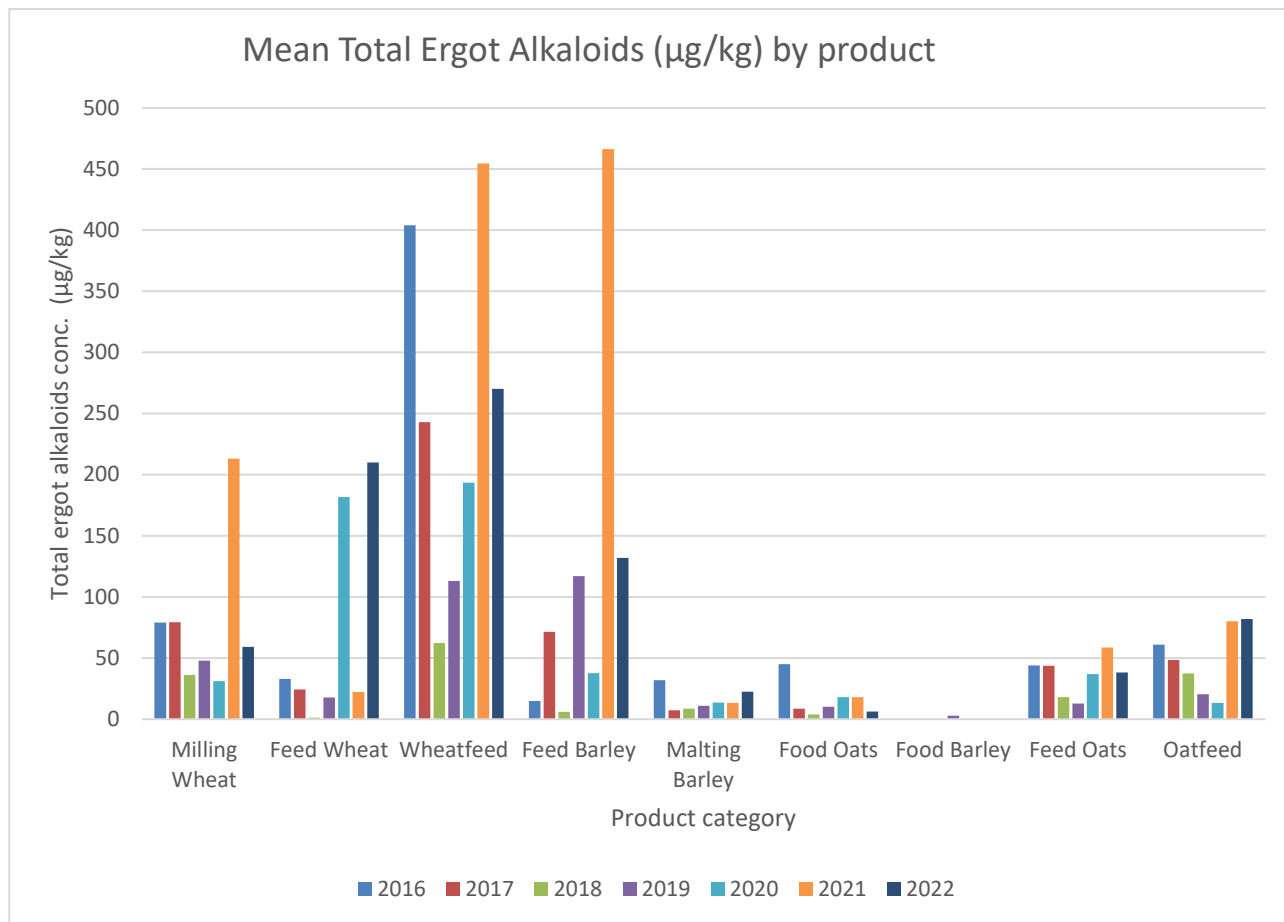
Table 10. Summarised sum total ergot alkaloid results ($\mu\text{g}/\text{kg}$) 2016–2018

	2018				2017				2016			
	>RL	Mean	Max	Med	>RL	Mean	Max	Med	>RL	Mean	Max	Med
	%	$\mu\text{g}/\text{kg}$			%	$\mu\text{g}/\text{kg}$			%	$\mu\text{g}/\text{kg}$		
Milling Wheat	42	36	765	<6.0	52	79.4	862	5.1	71	79	1435	6
Feed Wheat	45	1.2	7.4	<6.0	45	24.3	140	<6.0	60	33	148	3
Wheatfeed	90	62.3	326	40.3	95	243	633	205	100	404	1086	372
Feed Barley	90	6.1	32.1	1.1	55	71.4	383	8.7	67	15	69	3
Malting Barley	65	8.6	122	1.6	30	7.4	63.1	<6.0	70	32	275	3
Food Oats	38	3.9	47.2	<6.0	48	8.6	97.8	<6.0	60	45	710	8
Food Barley	n/a	n/a	n/a	n/a	0	<6.0	<6.0	<6.0	n/a	n/a	n/a	n/a
Feed Oats	85	18.1	159	3.2	18	43.6	407	<6.0	70	44	171	2
Oatfeed	100	37.5	263	11.6	100	48.4	111	43.1	100	61	160	61

Table 11. Summarised sum total ergot alkaloid results ($\mu\text{g}/\text{kg}$) 2019–2022

	2022				2021				2020				2019			
	>RL	Mean	Max	Med	>RL	Mean	Max	Med	>RL	Mean	Max	Med	>RL	Mean	Max	Med
	%	$\mu\text{g}/\text{kg}$			%	$\mu\text{g}/\text{kg}$			%	$\mu\text{g}/\text{kg}$			%	$\mu\text{g}/\text{kg}$		
Milling Wheat	42	59.1	961	<6.0	76	213	2603	<6.0	39	31.1	468	<6.0	46	47.8	429	<6.0
Feed Wheat	43	210	2802	<6.0	80	22	123	1.4	90	182	1542	31.1	60	17.7	100	8.2
Wheatfeed	100	270	865	204	100	454	1119	340	100	193	420	162	100	113	248	104
Feed Barley	47	132	1087	<6.0	93	466	6037	17	70	37.6	213	4.6	50	117	777	0.5
Malting Barley	23	22.5	304	<6.0	55	17.2	236	0.7	54	13.5	251	0.7	55	11	64	2.9
Food Oats	28	6.4	108	<6.0	14	10.3	232	<6.0	48	18.1	242	<6.0	38	10.1	59.1	<6.0
Food Barley	0	<6.0	<6.0	<6.0	0	<6.0	<6.0	<6.0	0	<6.0	<6.0	<6.0	100	2.8	2.8	2.8
Feed Oats	33	38.2	49	<6.0	100	58.7	151	45.1	60	37	258	7.4	40	12.9	107	<6.0
Oatfeed	83	81.9	143	<6.0	100	80	187	69.3	80	13.2	43	4.6	100	20.3	43.9	9.4

Figure 5. Mean Ergot Alkaloid levels in harvest samples



6.5. Ochratoxin A

Incidence, mean, maximum and median values for all commodities over the 7 years are displayed in tables 12 and 13 with mean total ochratoxin A levels displayed in Figure 6.

Incidence levels have remained relatively low throughout the project with only wheatfeed and oatfeed having consistently high (>75%) incidence. Mean and median levels have also been consistently low for all product types; therefore, showing that the overwhelming majority of samples received have either been below or close to the method RL.

Throughout the 7 years of the project, there have only been 4 instances of ML exceedances. One sample of food oats in 2017, 2022 and 2023 and one sample of milling wheat in 2023, showing that OTA is well controlled.

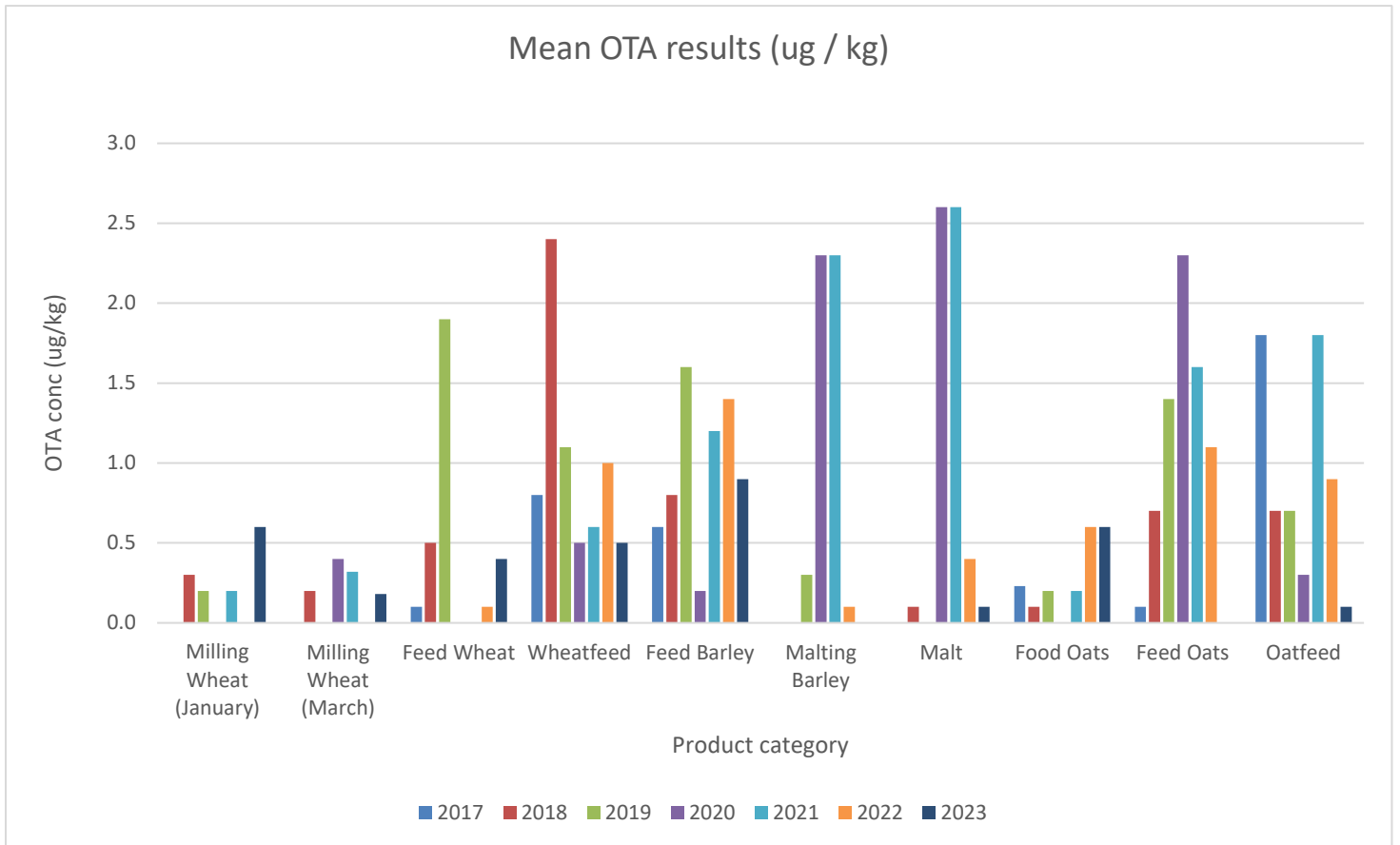
Table 12. Ochratoxin A levels ($\mu\text{g}/\text{kg}$) 2017–2019

	2017				2018				2019			
	>RL	Mean	Max	Med	>RL	Mean	Max	Med	>RL	Mean	Max	Med
	%	$\mu\text{g}/\text{kg}$			%	$\mu\text{g}/\text{kg}$			%	$\mu\text{g}/\text{kg}$		
Milling Wheat (Jan)	3	0.03	1	<0.8	25	0.3	3.9	<0.2	16	0.2	2.8	<0.2
Milling Wheat (March)	6	0.02	0.4	<0.2	25	0.2	4	<0.2	n/a	n/a	n/a	n/a
Feed Wheat	13	0.1	1.4	<0.2	40	0.5	4.8	<0.2	15	1.9	72.5	<0.2
Wheatfeed	91	0.8	2.1	0.7	10	2.4	11	1.5	60	1.1	5.4	0.3
Feed Barley	14	0.6	14.6	<0.2	30	0.8	9.2	<0.2	13	1.6	28.4	<0.2
Malting Barley	0	<0.2	<0.2	<0.2	5	0.3	6.3	<0.2	10	<0.2	2.3	<0.2
Malt	15	0.1	1.5	<0.2	5	<0.2	0.3	<0.2	25	0.2	2.6	<0.2
Food Oats	7	0.23	5.7	<0.2	27	0.1	0.8	<0.2	7	0.2	4.8	<0.2
Feed Oats	20	0.1	0.9	<0.2	20	0.7	7	<0.2	20	1.4	13.3	<0.2
Oatfeed	70	1.8	7.5	1.1	88	0.7	1.9	0.4	80	0.7	1.9	0.8

Table 13. Ochratoxin A levels ($\mu\text{g}/\text{kg}$) 2020–2023

	2020				2021				2022				2023			
	>RL	Mean	Max	Med	>RL	Mean	Max	Med	>RL	Mean	Max	Med	>RL	Mean	Max	Med
	%	$\mu\text{g}/\text{kg}$			%	$\mu\text{g}/\text{kg}$			%	$\mu\text{g}/\text{kg}$			%	$\mu\text{g}/\text{kg}$		
Milling Wheat (Jan)	12	<0.2	4.1	<0.2	12	0.2	3.5	<0.2	0	<0.2	<0.2	<0.2	20	0.6	8.7	<0.2
Milling Wheat (March)	19	0.4	4	<0.2	12	0.32	7.4	<0.2	8	<0.2	0.5	<0.2	8	0.18	4	<0.2
Feed Wheat	5	<0.2	0.6	<0.2	13	<0.2	4.8	<0.2	13	0.1	2.6	<0.2	40	0.4	11	<0.2
Wheatfeed	90	0.5	0.7	0.6	67	0.6	2.1	0.5	92	1	3.2	<0.2	42	0.5	2	<0.2
Feed Barley	10	0.2	6.2	<0.2	18	1.2	17.2	<0.2	8	1.4	49.7	<0.2	33	0.9	21.1	<0.2
Malting Barley	10	<0.2	2.3	<0.2	n/a	n/a	n/a	n/a	10	0.1	1.5	<0.2	5	<0.2	0.3	<0.2
Malt	25	0.2	2.6	<0.2	n/a	n/a	n/a	n/a	20	0.4	6.2	<0.2	45	0.1	1.1	<0.2
Food Oats	20	<0.2	0.6	<0.2	13	0.2	4	<0.2	7	0.6	17.2	<0.2	7	0.6	17.6	<0.2
Feed Oats	30	2.3	22.5	<0.2	13	1.6	12.4	<0.2	50	1.1	2.9	<0.2	0	<0.2	<0.2	<0.2
Oatfeed	67	0.3	0.6	<0.2	100	1.8	4.2	1.1	100	0.9	1.5	0.95	33	0.1	0.5	<0.2

Figure 6. Mean Ochratoxin A results ($\mu\text{g}/\text{kg}$)



6.6. Metals (Milling Wheat and Food Oats)

Throughout the 7 years of the project, only milling wheat and food oats have metals data spanning more than 4 years. Therefore, those results are displayed in Tables 14, 15 and 16 and Figures 7 and 8.

Incidence levels of arsenic and lead were low throughout the project with maximum levels found only marginally above the RL. Mercury was not detected in milling wheat or food oats throughout the project.

Cadmium was detected in the majority of samples during the project, with the lowest incidence rate being 33% in food oats (2019). The mean and maximum concentrations measured showed very little variance throughout the 7 years and no ML exceedances were found [7].

As part of ongoing monitoring, aluminium, copper and nickel were also included in the suite of metals analyses each year. Incidence levels for these three elements was almost 100% every year. Some values for aluminium were 'high' (food oats in 2021 – 148.9 mg/kg, milling wheat in 2020 -101 mg/kg), however, these were atypical samples and did not reflect the low levels generally observed during the project.

Table 14. Metals concentrations (mg/kg) in Milling wheat 2017–2019

	2017				2018				2019 (not requested)			
	>RL	Mean	Max	Med	>RL	Mean	Max	Med	>RL	Mean	Max	Med
	%	mg/kg			%	mg/kg			%	mg/kg		
Al	97	4.71	37.6	2.7	96	6	32	3.1				
Ni	100	0.12	0.3	0.1	100	0.2	0.32	0.16				
Cu	100	3.27	4.5	3.2	100	3.6	5.2	3.7				
As	23	<0.01	0.02	<0.01	30	0.02	0.08	0.01				
Cd	100	0.04	0.07	0.04	98	0.04	0.1	0.04				
Hg	0	<0.01	<0.01	<0.01	0	<0.01	<0.01	<0.01				
Pb	13	<0.01	0.03	<0.01	28	0.02	0.03	0.01				

Table 15. Metals concentrations (mg/kg) in Milling wheat 2020–2022

	2020				2021				2022			
	>RL	Mean	Max	Med	>RL	Mean	Max	Med	>RL	Mean	Max	Med
	%	mg/kg			%	mg/kg			%	mg/kg		
Al	96	10.8	101	4.7	96	11.5	94.4	5.2	100	3.72	12.2	2.8
Ni	100	0.31	1.21	0.17	100	0.27	1.34	0.2	100	0.18	0.44	0.17
Cu	100	3.41	4.4	3.2	100	3.7	5.7	3.6	100	3.52	5.5	3.5
As	16	0.01	0.04	<0.01	28	0.01	0.07	<0.01	24	<0.01	0.03	<0.01
Cd	100	0.05	0.14	0.04	100	0.04	0.09	0.04	100	0.039	0.09	0.04
Hg	0	<0.01	<0.01	<0.01	0	<0.01	<0.01	<0.01	0	<0.01	<0.01	<0.01
Pb	32	0.01	0.05	<0.01	32	0.01	0.12	<0.01	16	<0.01	0.09	<0.01

Figure 7. Mean levels of metals (mg/kg) in milling wheat

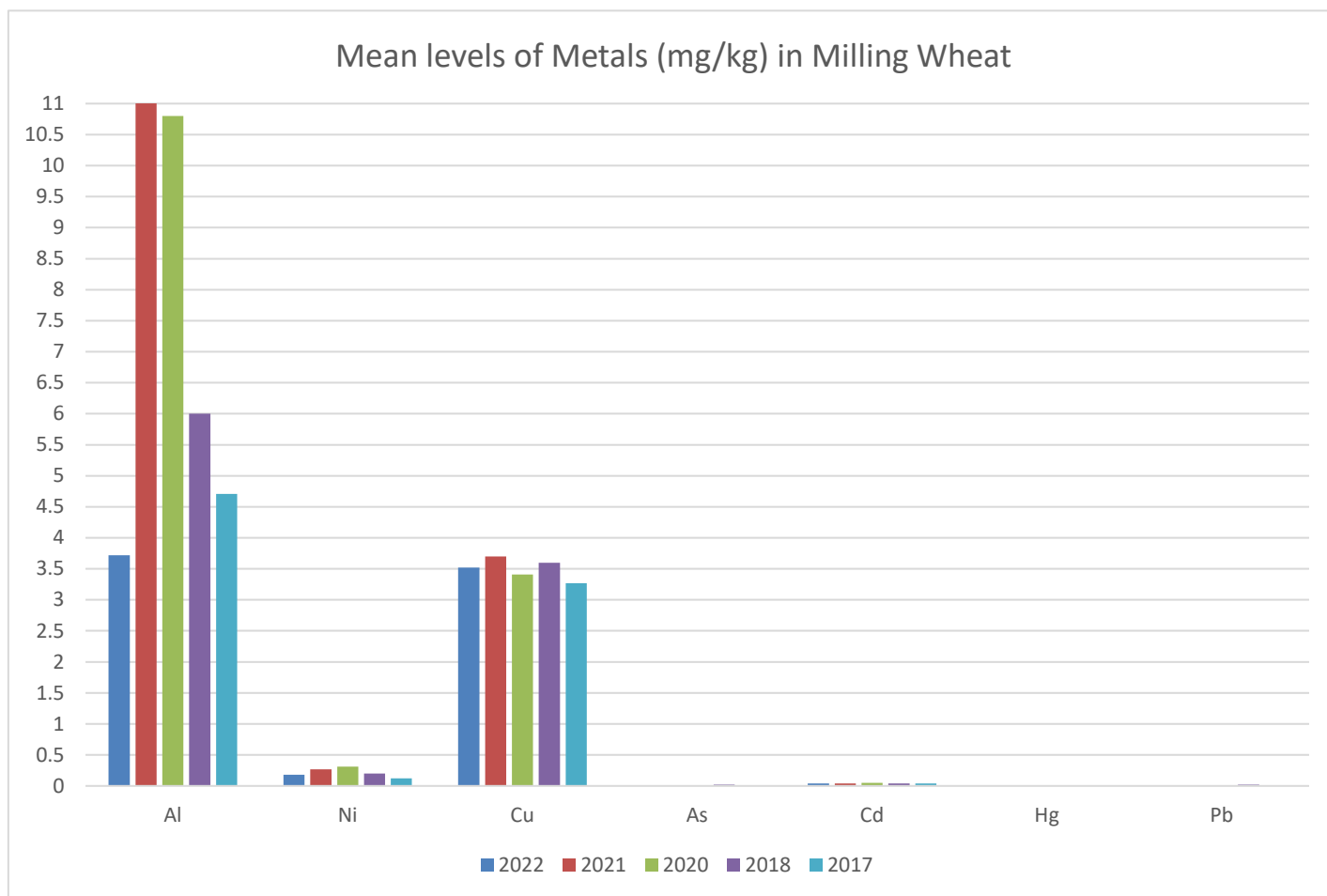
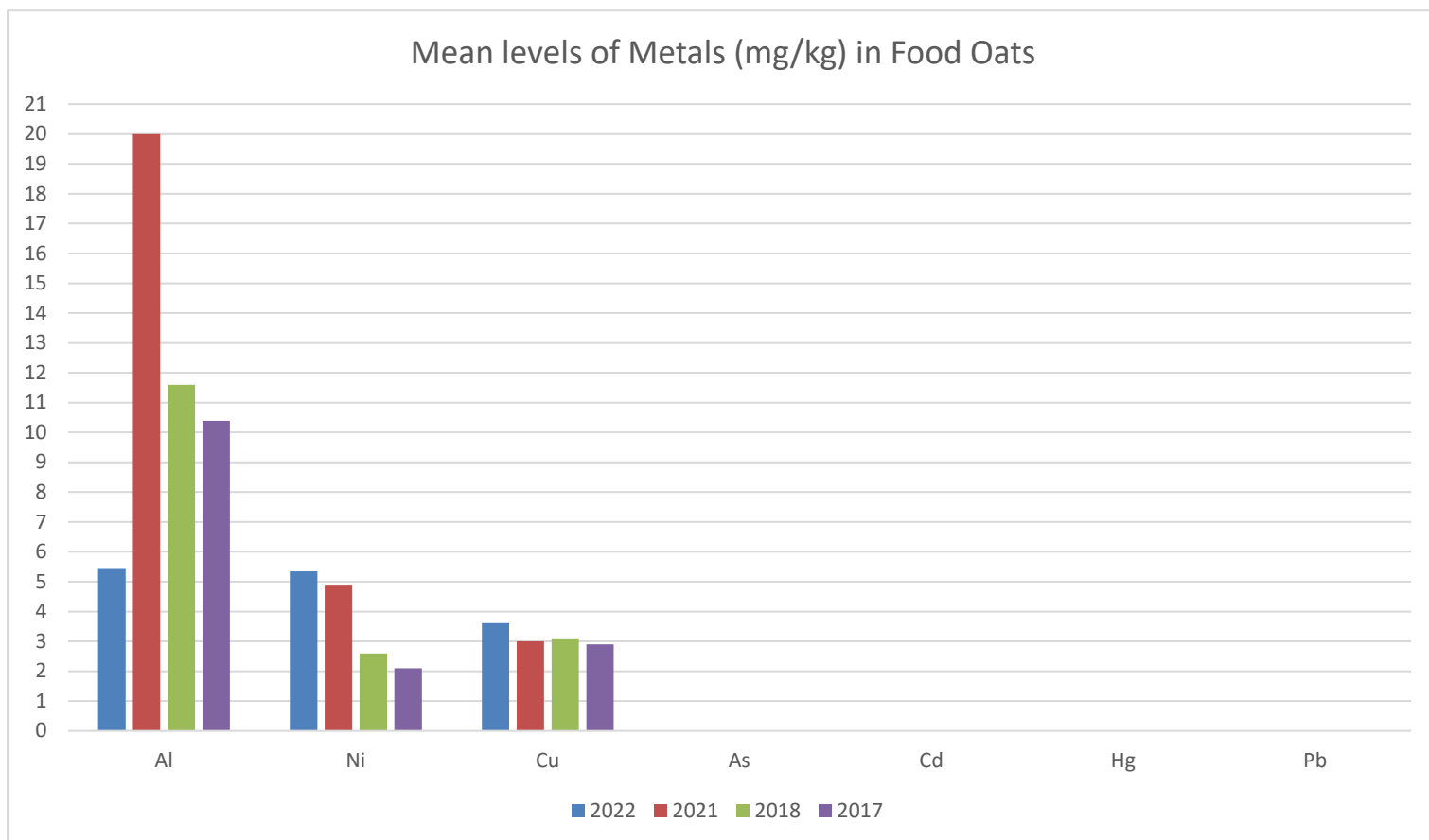


Table 16. Metals concentrations (mg/kg) in Food oats 2016–2022

	2022				2021				2018				2016			
	>RL	Mean	Max	Med	>RL	Mean	Max	Med	>RL	Mean	Max	Med	>RL	Mean	Max	Med
	%	mg/kg			%	mg/kg			%	mg/kg			%	mg/kg		
Al	100	5.46	20.1	3.65	100	20.0	148.9	2.5	100	11.6	76.7	6.1	100	10.39	37.5	7.1
Ni	100	5.35	7.75	5.10	100	4.9	8.35	4.47	100	2.6	5.5	2.5	100	2.1	3.77	1.95
Cu	100	3.61	4.5	3.60	100	3.0	3.6	3.1	100	3.1	4.2	3	100	2.9	3.7	2.9
As	57	0.01	0.03	0.01	30	0.02	0.06	<0.01	33	0.01	0.04	<0.01	53	0.01	0.03	0.01
Cd	71	0.02	0.03	0.02	70	0.01	0.03	0.01	63	0.02	0.05	0.01	33	0.01	0.03	<0.01
Hg	0	<0.01	<0.01	<0.01	0	<0.01	<0.01	<0.01	0%	<0.01	<0.01	<0.01	0	<0.01	<0.01	<0.01
Pb	21	<0.01	0.02	<0.01	50	0.02	0.08	0.01	80	0.02	0.11	0.01	60	0.01	0.04	0.01

Figure 8. Mean levels of metals (mg/kg) in food oats



6.7. Co-occurrence data – Mycotoxins

Full tables covering all 7 years of the project can be found in appendix 2.

Fera’s approach was to produce heatmaps for all samples submitted over the 7 years of the project. The aim was to show the distribution of results from year to year in an easily comparable pattern (in much the same way as a QR code) to provide a snapshot of mycotoxins occurrence from year to year.

Various parameters were set to distinguish a value below the RL (white) to those above the RL with the colour (blue) increasing in intensity the higher the concentration value is.

The following limits were applied for the charts to be created.

BOBMA - Oat samples

(1 Food Barley sample is present in charts 2017 to 2022, not in 2016)

DON – 1750 µg/kg (Used for DON to NEO, and DON-3-GLc).

ZEN – 100 µg/kg (Used for ZEN to β-ZEL-14-GLc).

Ergot Alkaloids – 100 µg/kg (All Ergot columns) - EU Regulations

HT-2 and T-2 – 1250 µg/kg - EU Regulations

AIC – Mixed product types

Oatfeed and Wheatfeed (Compound feed regs).

DON – 5 mg/kg

ZEN - 0.5 mg/kg

Feed Wheat, Barley and Oats (Feed materials regs).

DON – 8 mg/kg

ZEN – 2 mg/kg

UKFM – Milling wheat

DON – 1250 µg/kg

ZEN – 100 µg/kg

Ergot Alkaloids – 100 µg/kg

HT-2 and T-2 – 50 µg/kg

MAGB – Malting Barley

DON – 1250 µg/kg

ZEN – 100 µg/kg

Ergot Alkaloids – 100 µg/kg

HT-2 and T-2 – 50 µg/kg

The main patterns observed are ones which would be expected, for example, where raised levels of DON/ZEN are measured, you are likely to detect levels of DON-3-Glucoside/ α -ZEL + β -ZEL.

Below are just some small observations made for co-occurrence for each partner.

AIC

For oat feed samples, where there are elevated levels of T-2+HT-2 raised levels of DON, NIV, DON-3-GLC, and T-2- α 3-GLC are more likely to occur. However, during some years of the study (e.g. 2020), 3Ac DON was also detected at raised levels, there are also examples of ZEN occurring in some samples when T-2 + HT-2 are raised but not in others.

BOBMA

The above pattern was also seen to exist in food oats. However, it cannot be stated with any real degree of confidence, as there are several examples of individual analytes being measured at high levels whilst other remain low. For example, in 2022, ZEN was detected at raised levels in a sample, however, this same sample had low levels of T-2+ HT-2. There was also an example of very high levels of T-2+HT-2 toxins being present, but DON was not detected above the RL.

UKFM

There is some evidence, over the 7 years, to suggest that where DON is measured at levels of >50 µg/kg, there is an increased likelihood of detecting DON-3-GLC. However, there is also evidence of DON-3-GLC being detected when DON was measured at low levels.

Overall, the distribution of mycotoxins for milling wheat does tend to be more sporadic (other than DON being detected regularly each year), with no obvious patterns of co-occurrence.

MAGB

Unlike in milling wheat, there doesn't seem to be any link between DON and DON-3-GLC in malting barley. Where DON is detected around 100 µg/kg and above, DON-3-GLC is not detected at all.

As per the patterns seen in food oats and oat feed, there are several examples of high T-2+HT-2 toxins levels, also detecting raised levels of NIV, DON and T-2- α3-GLC. However, there are also several examples of this pattern not occurring.

There are also examples in malting barley of individual mycotoxins being detected at high levels, but no other mycotoxins detected above the RL.

For all product types, there is no clear and obvious pattern of co-occurrence seen throughout the 7 years of the project. There is no evidence to suggest that if residue X occurs then so will residue Y. Any raised levels of ergot alkaloids do not suggest raised levels of fusarium toxins and vice versa.

AHDB have also carried out their own statistical analysis of co-occurrence in fusarium toxins over the course of the project.

6.8. Pesticides

Pesticides data from the 7-year contaminants monitoring project has been disseminated and is presented below to discern trends in the usage of pesticides over the period 2016-2023.

As for mycotoxins, samples were tested both fresh from harvest and after storage. Different testing regimes were undertaken for fresh harvest and stored samples.

As there is a large volume of data, only pertinent results reported with positive values have been included in this report. Of this dataset, we have looked at the core set of analytes in their categories and applied those as core compounds from all sampling periods. This has been done as not all core compounds are consistently sought in each sampling period. By doing this, we are

able to provide more consistent and meaningful data in identifying year on year trends. In the case of trinexapac-ethyl and chlorate, the data has been omitted from the analysis, as these compounds were not sought consistently across the 7-year period; therefore, no trends for these compounds can be ascertained and inclusion in the analysis would skew any trends in the non-core compounds data.

6.8.1. Total number of pesticide residues – harvest samples 2016–2022

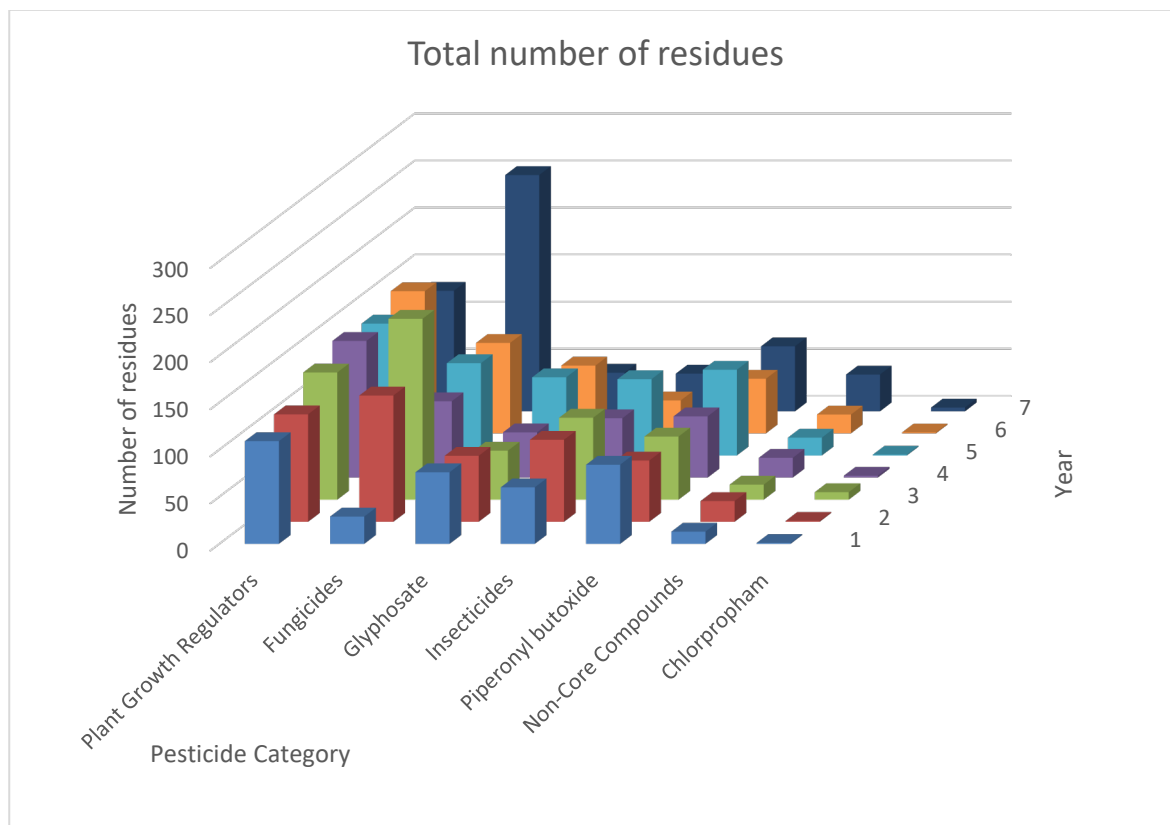
The following table (Table 17) shows the total number of residues found in each pesticides category over the 7-year sampling period for fresh harvest samples collected in September (approximately) each year from 2016-2022.

Table 17. Total number of pesticide residues found in each category, 2016–2022

Year	PGRs	Fungicides	Glyphosate	Insecticides	Piperonyl butoxide	Non-Core Cmpds	Chlorpropham
1	109	29	76	60	84	13	1
2	114	134	70	87	65	22	1
3	135	192	52	87	67	16	8
4	145	81	48	63	65	21	2
5	140	98	83	81	91	19	1
6	151	96	72	35	58	20	1
7	128	251	41	40	69	39	4

From the information contained in Table 17 above, the following 3-D chart (Figure 9) showing the total number of residues per category over the 7-year period has been created to visualise the data more easily.

Figure 9. Total number of pesticide residues found per category, fresh harvest 2016–2022



From the chart (Figure 9) above the following can be discerned:

- The use of plant growth regulators is relatively consistent year on year.
- The use of fungicides varies from year to year with Year 3 (Harvest Year 2018) and Year 7 (harvest Year 2022) standing out with particularly high incidences of residues detected.
- Glyphosate is frequently found in each year period with some variation but a relatively low degree of variance.
- The use of insecticides is consistent across the years with Year 6 (Harvest Year 2021) and Year 7 (Harvest Year 2022) both returning a significantly lower incidence of residues detected.
- Piperonyl butoxide is frequently found in each year period with some variation but a relatively low degree of variance.
- Non-core compounds were detected consistently across the sampling period with Year 7 (Harvest Year 2022) being the outlier and approximately double the number of detections compared to previous years.
- Chlorpropham has been consistently detected across the years in low numbers. Year 3 (Harvest Year 2018) and Year 7 (Harvest Year 2022) show the highest number of residues detected. Chlorpropham residues are thought to be from contamination from storage rather than misuse.

The following charts (Figure 10 to Figure 16) represent the total number of residues detected per year in individual categories:

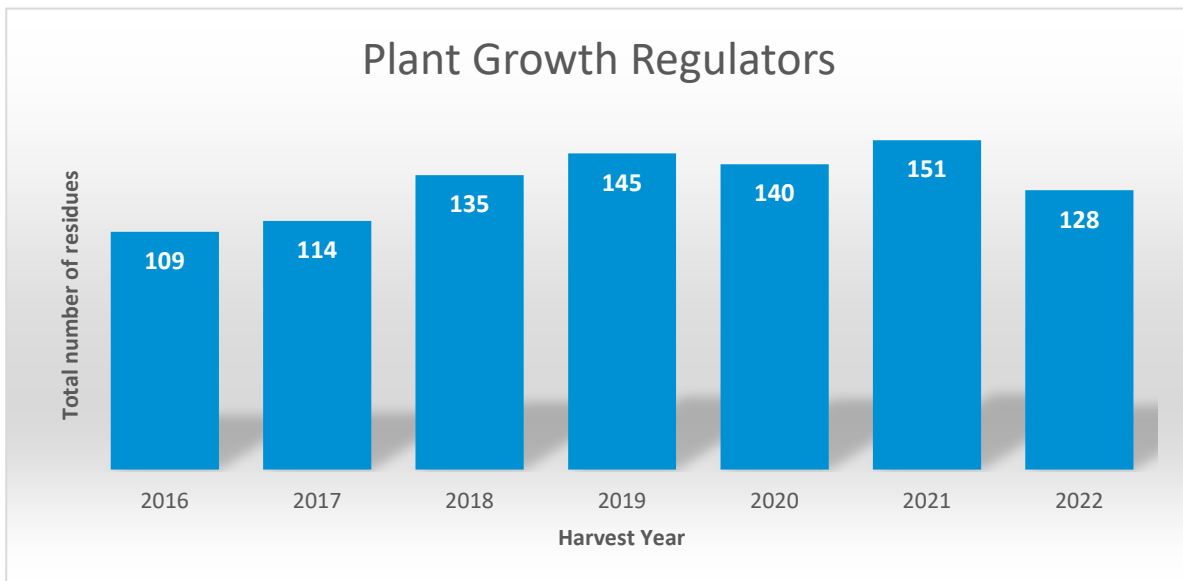


Figure 10. Plant Growth Regulators – Harvest samples 2016–2022

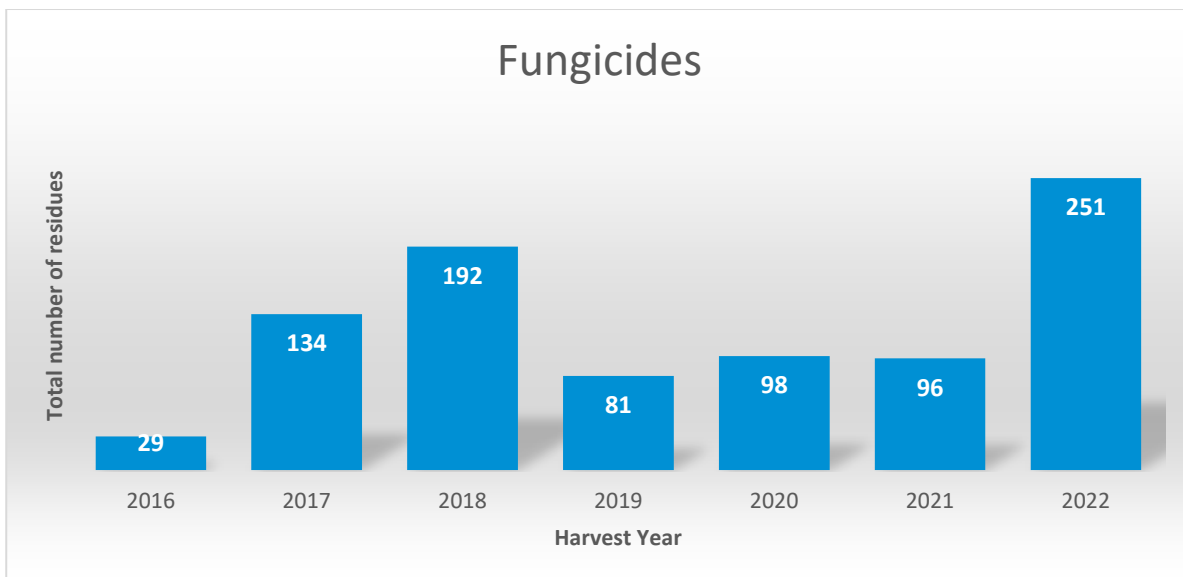


Figure 11. Fungicides – Harvest samples 2016–2022

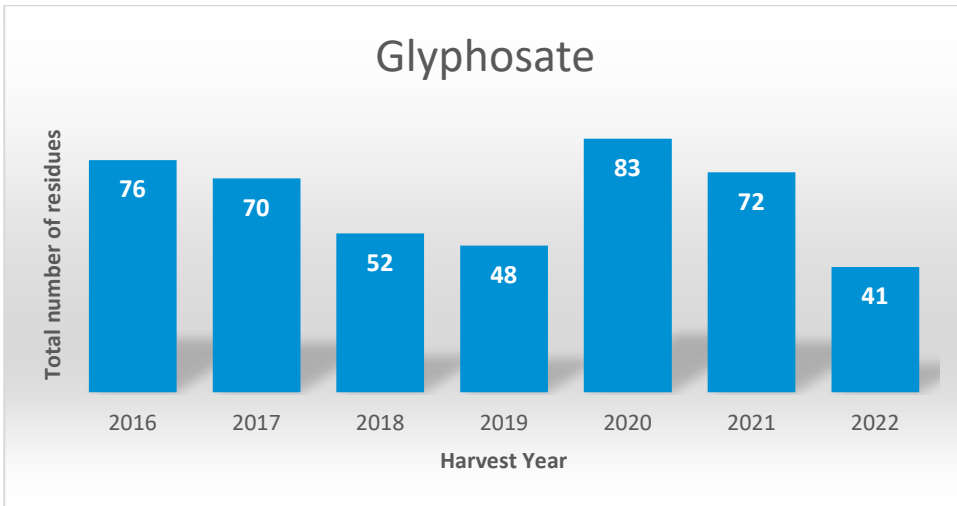


Figure 12. Glyphosate - Harvest samples 2016–2022

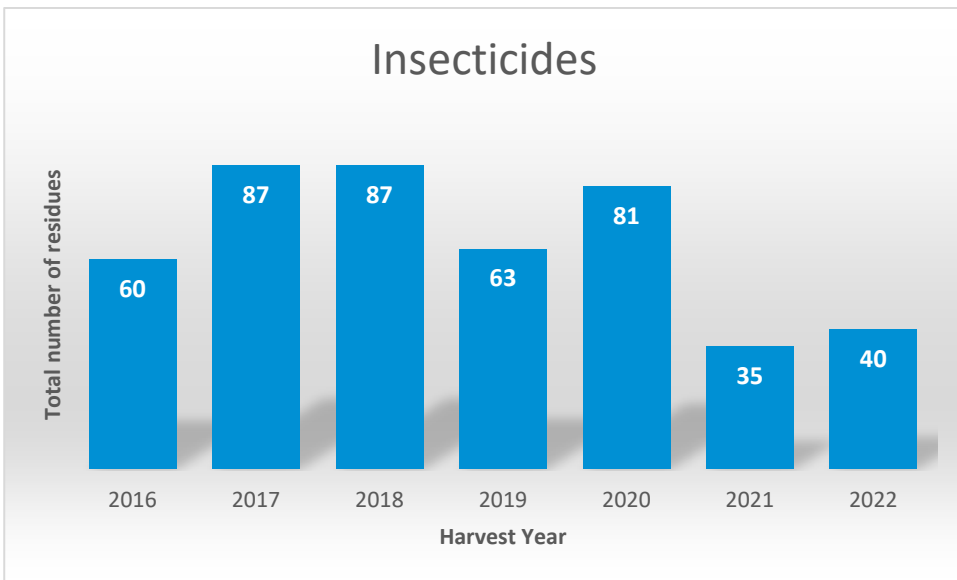


Figure 13. Insecticides - Harvest samples 2016–2022

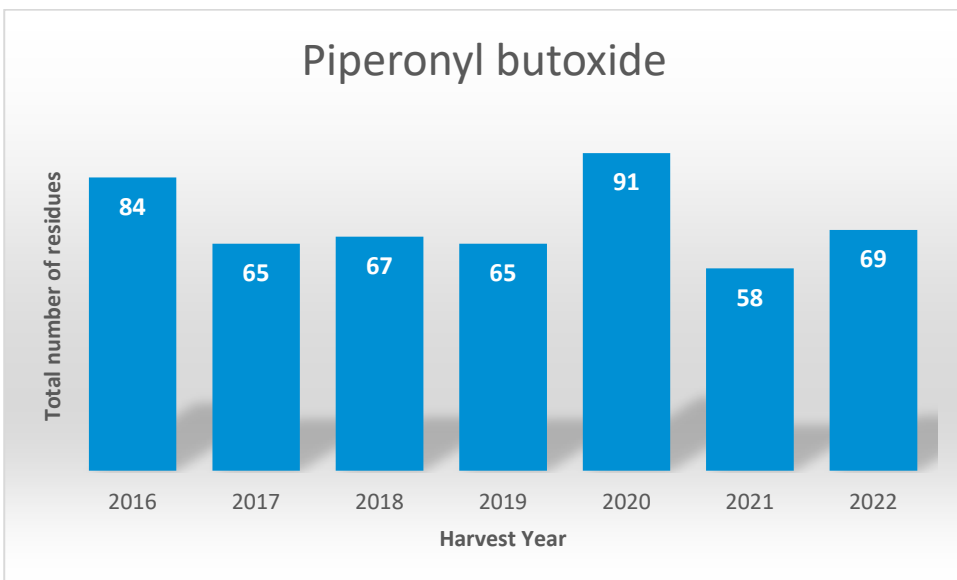


Figure 14. Piperonyl butoxide - Harvest samples 2016–2022

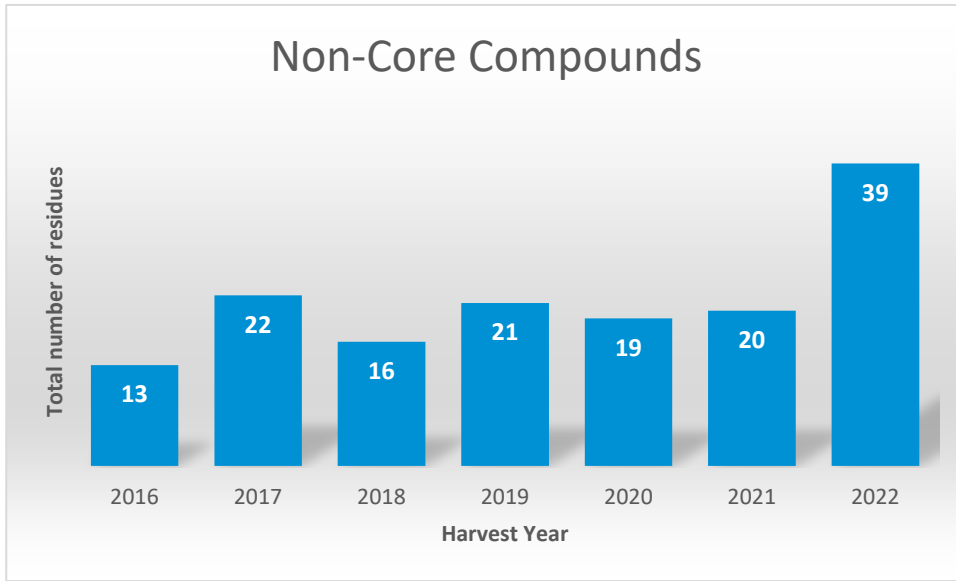


Figure 15. Non-core compounds - Harvest samples 2016–2022

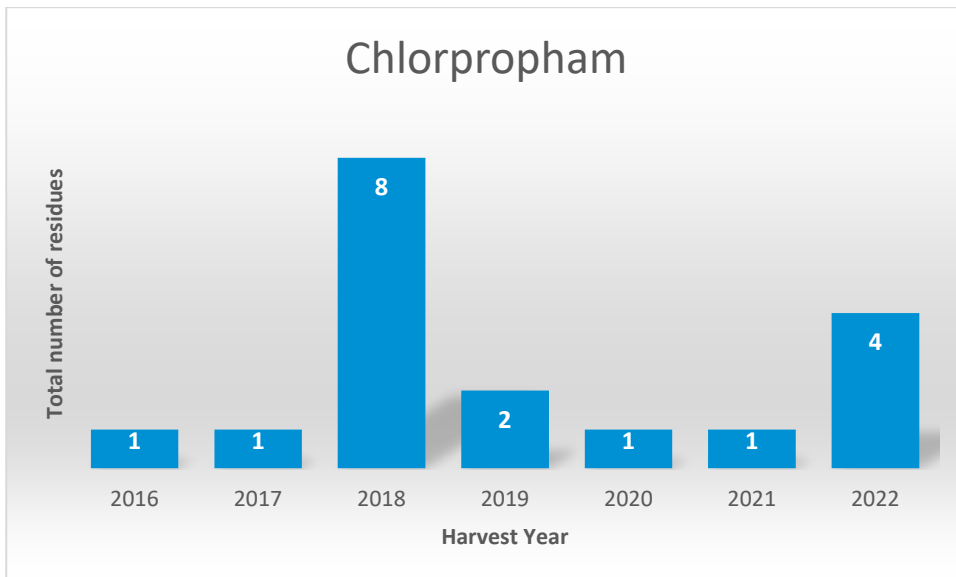


Figure 16. Chlorpropham - Harvest samples 2016–2022

6.8.2. Total number of samples tested for stored samples 2016–2023

As described above stored samples were collected at different time points. Sampling was due to take place in the period November to the following March but often started and finished later than these dates. The total number of samples tested in each category per year for stored samples is consistent across the sampling period. The numbers are tabulated below in Table 18.

Table 18. Total number of stored samples tested in each category, 2016–2023

Year	PGRs	Fungicides	Glyphosate	Insecticides	Piperonyl butoxide	Non-Core Cmpds	Chlorpropham
1	121	322	150	351	322	351	351
2	124	321	157	344	321	344	344
3	120	315	154	340	315	340	340
4	120	315	150	340	315	340	340
5	121	313	151	339	313	339	339
6	121	316	157	341	316	341	341
7	122	317	156	342	317	342	342

From the information contained in Table 18 above, the following 3-D chart (Figure 17) showing the average number of residues per category over the 7-year period has been created to visualise the data more easily.

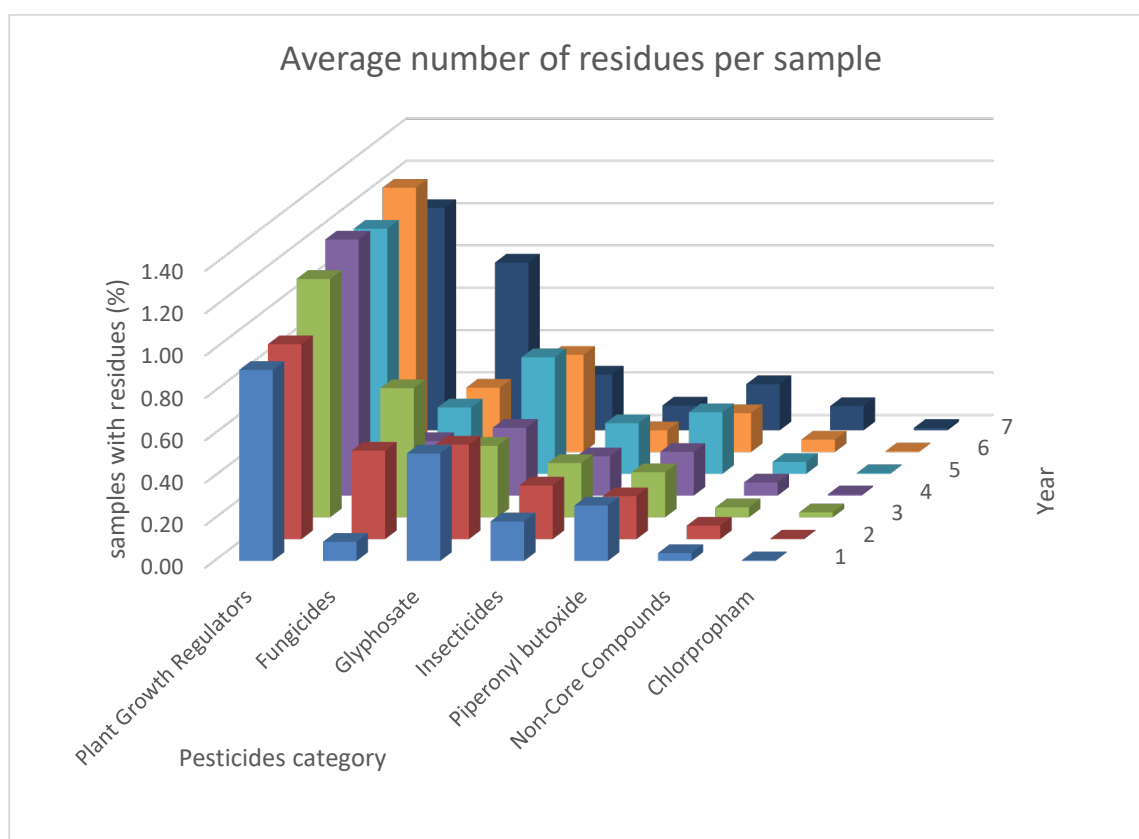


Figure 17. Average number of pesticides residues per category for stored samples, 2016–2023

From the chart (Figure 17) above, the following can be discerned:

- Plant growth regulators show the highest average number of residues per sample. Many samples have both chlormequat and mepiquat residues which has pushed the average number of residues per sample above 1, although some samples do not contain residues of plant growth regulators.
- The average number of fungicides per sample varies from year to year with Year 3 (Harvest Year 2018), and Year 7 (harvest Year 2022) standing out with higher average number of residues per sample detected. A pattern which mirrors the total number of fungicide residues per year.
- Glyphosate on average is the second most common average residue per sample and is consistent over the sampling period with slight annual variations.
- Average residues of insecticides per sample are relatively consistent between sampling periods with Year 6 (Harvest Year 2021) and Year 7 (Harvest Year 2022) both returning a significantly lower incidence of residues detected mirroring the total number of residues found profile.
- Piperonyl butoxide average residues per sample are very consistent between the sampling periods with little variation.
- The average number of non-core compounds residues per sample detected were consistent across the sampling period except in Year 7 (Harvest Year 2022), which was the outlier with approximately double the average number compared to previous years.
- Chlorpropham average residues per sample were very small due to the low number of incidences detected. Year 3 (Harvest Year 2018), and Year 7 (Harvest Year 2022) had the highest number of average residues per sample.

6.8.3. Average number of residues detected for stored samples, 2016–2023

The following charts (Figure 18 to Figure 24) represent the average number of residues per sample detected per year in individual categories.

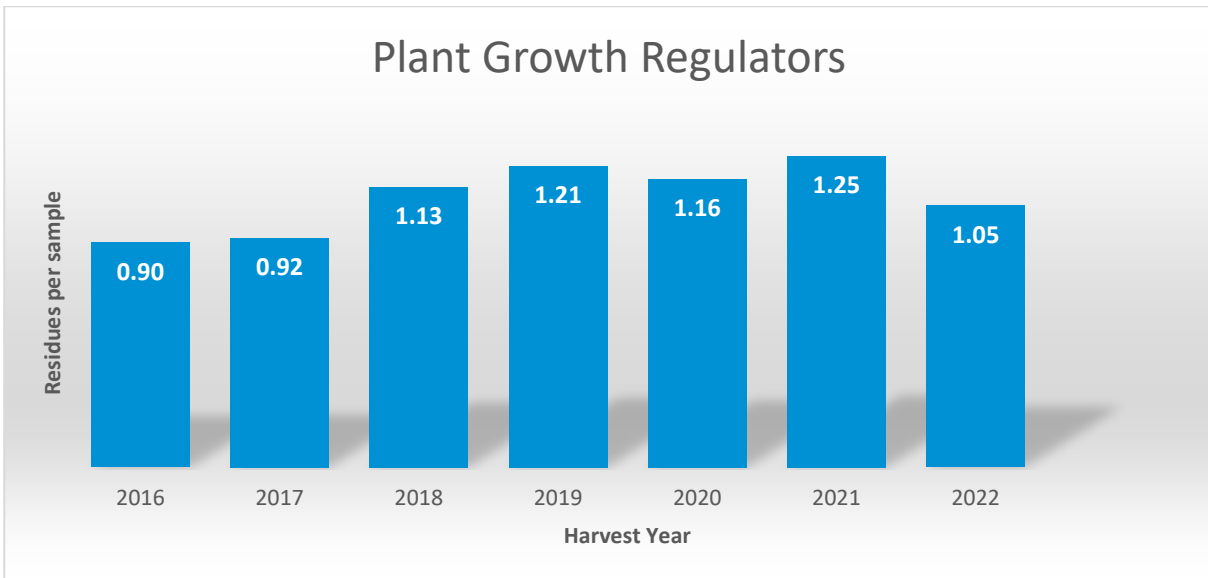


Figure 18. Plant Growth Regulators – Stored samples 2016–2023

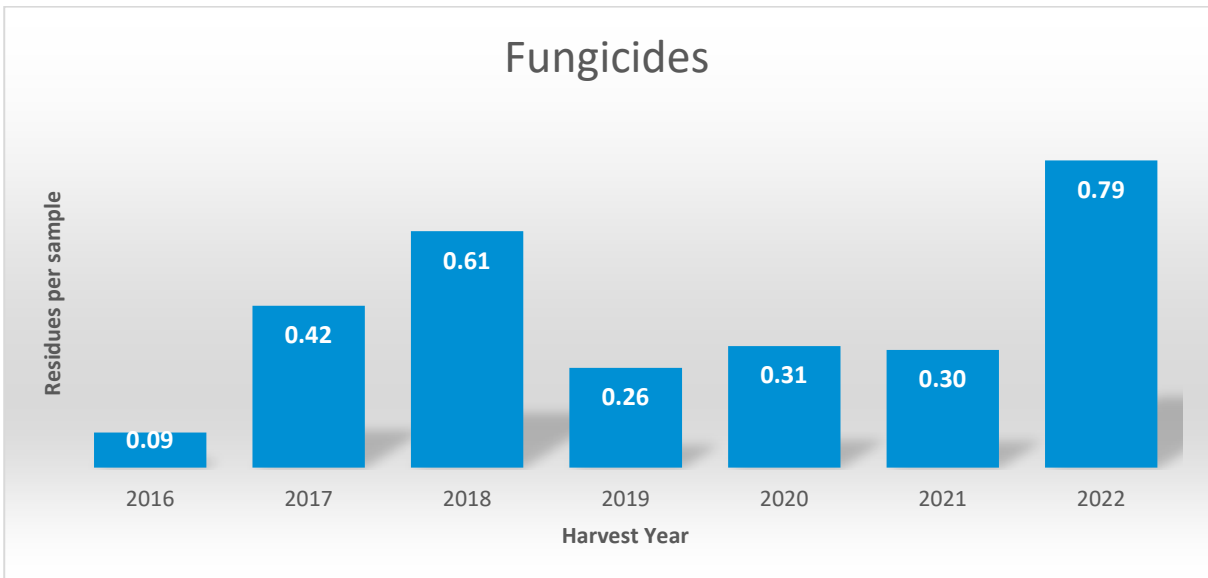


Figure 19. Fungicides – Stored samples 2016–2023

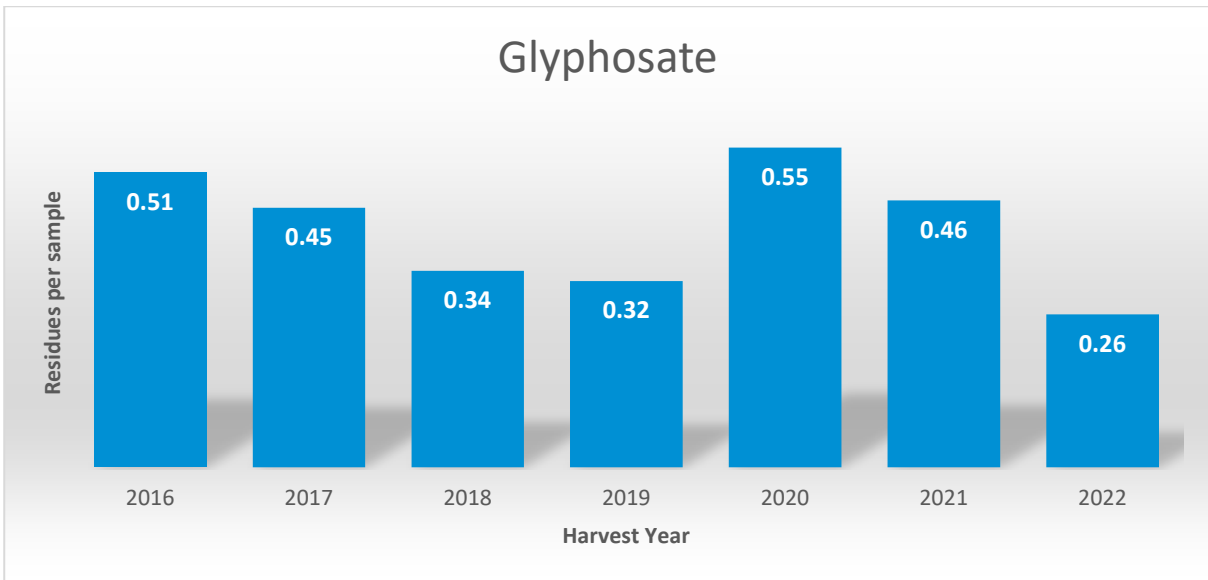


Figure 20. Glyphosate – Stored samples 2016–2023

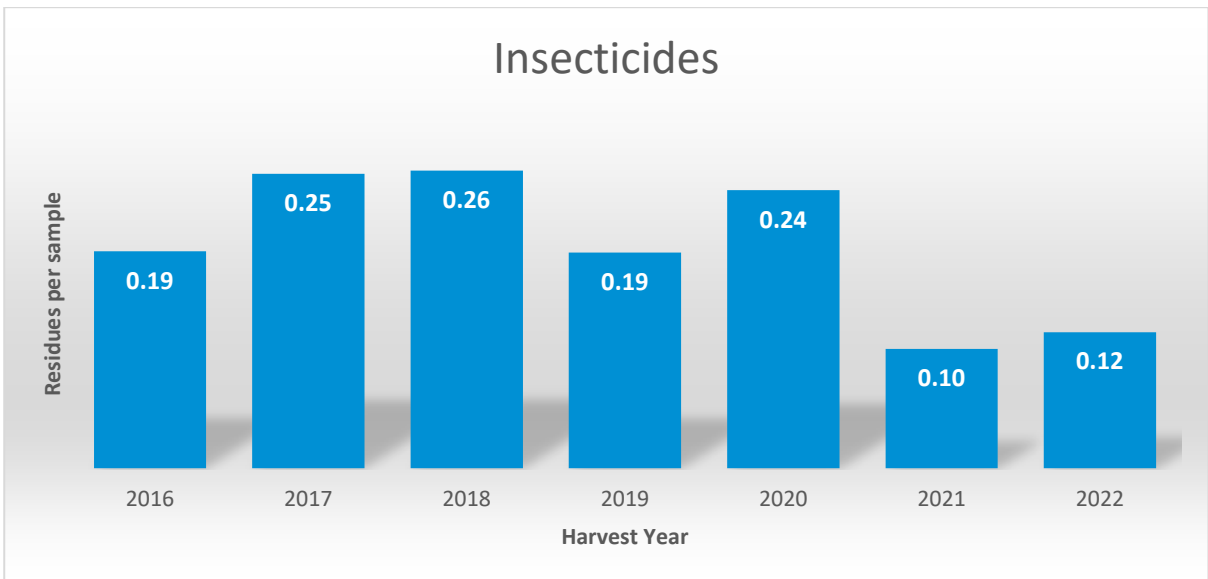


Figure 21. Insecticides – Stored samples 2016–2023

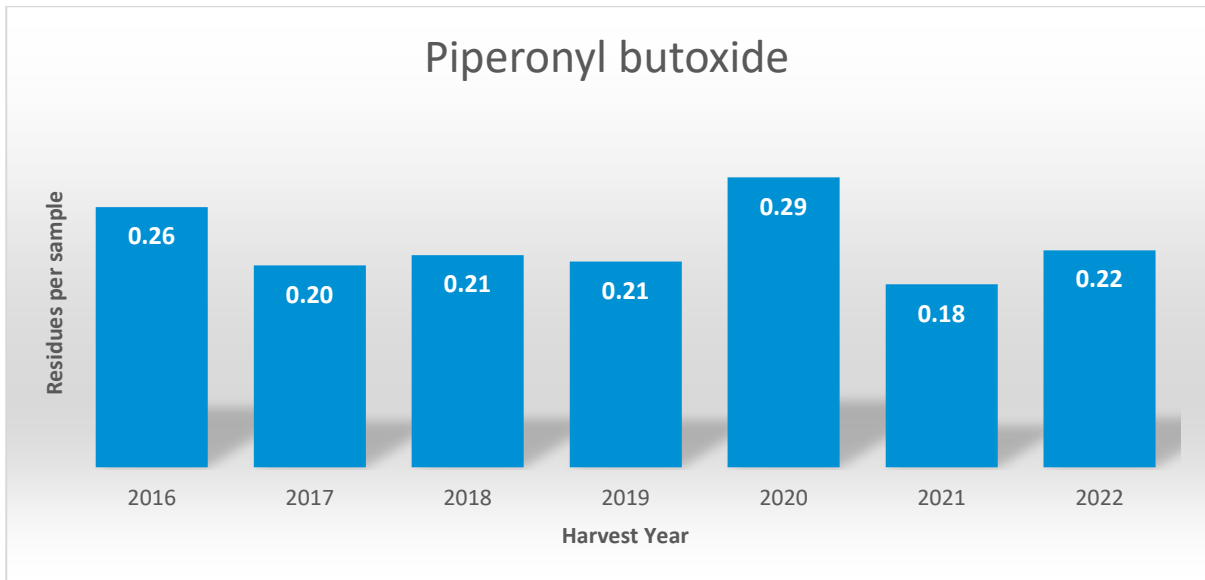


Figure 22. Piperonyl butoxide – Stored samples 2016–2023

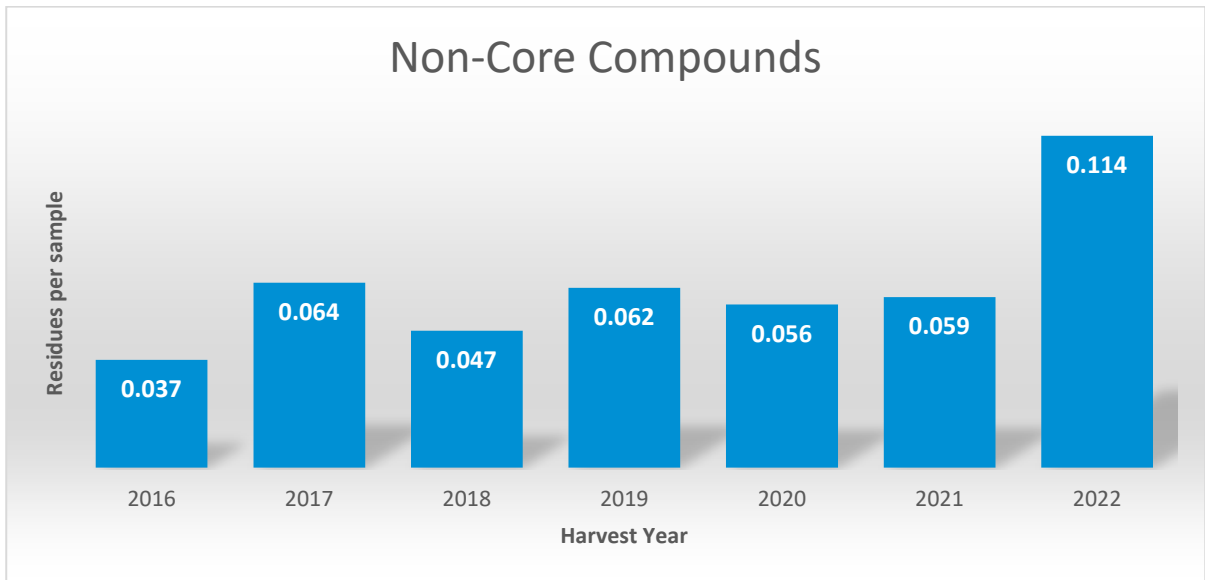


Figure 23. Non-Core Compounds – Stored samples 2016–2023

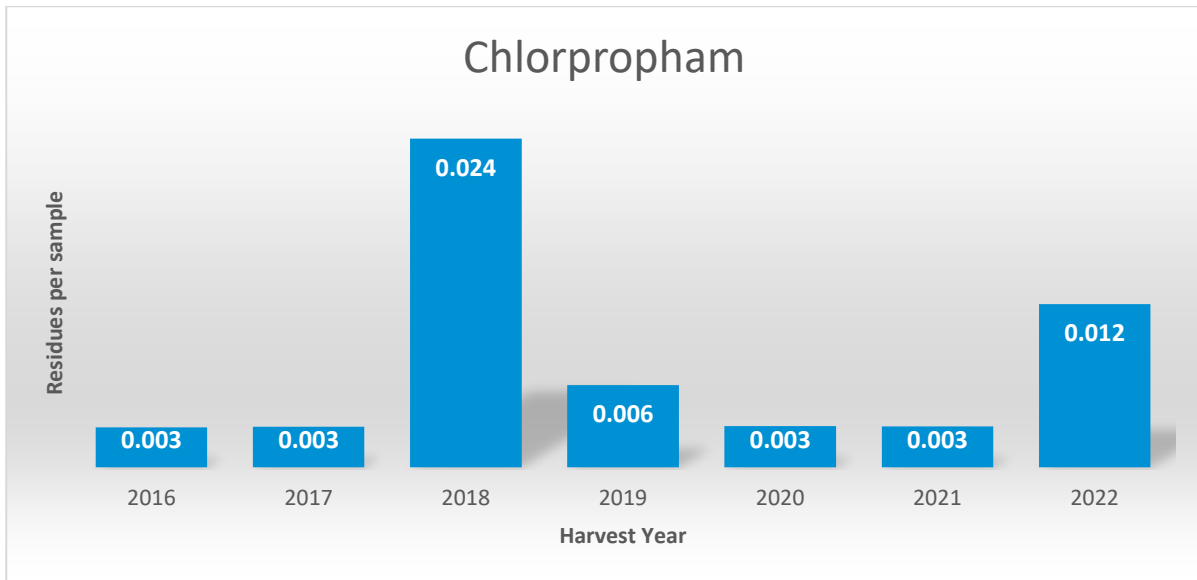


Figure 24. Chlorpropham – Stored samples 2016–2023

6.8.4. Pesticide frequency:

Data from the 7-year study provides an insight into the frequency of residues on a per sample basis. 2625 samples were tested for the various requirements with 1598 samples containing 1 or more residues. Table 19 below shows the distribution of the frequency of residues from the total number of samples tested. Figure 25 below shows the frequency distribution in an easier to visualise pie chart.

Table 19. Frequency of distribution of residues per sample

Residues per individual sample	Number of samples	Percentage of total samples analysed
0	1027	39%
1	669	25%
2	474	18%
3	261	10%
4	132	5%
5	42	2%
6+	20	1%

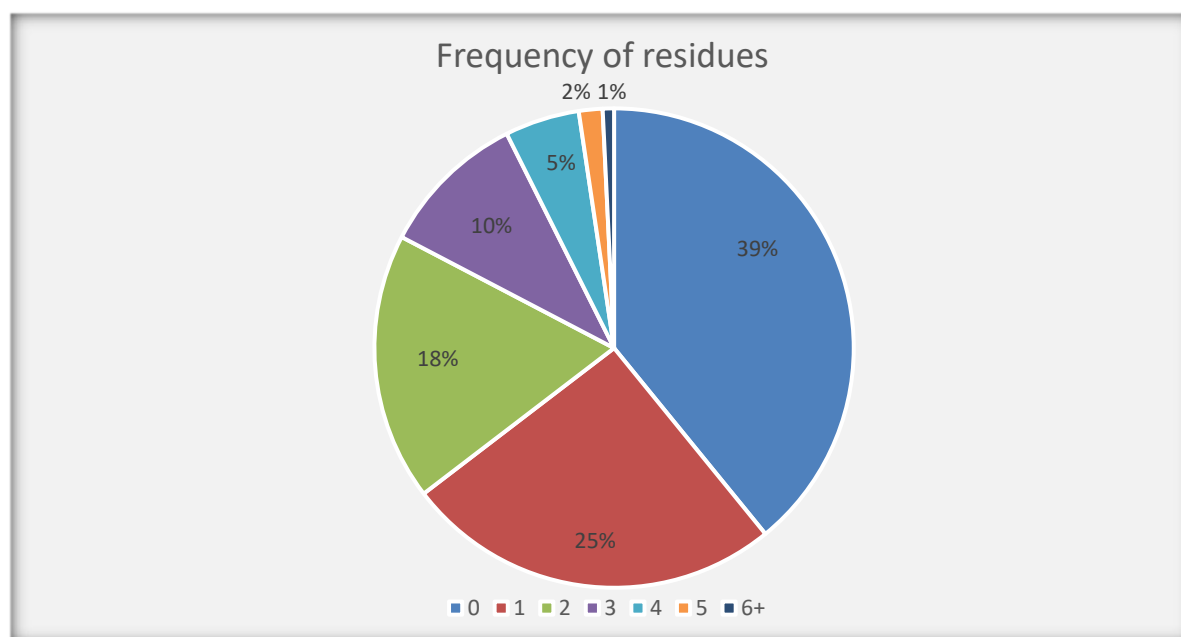


Figure 25. Pie chart of frequency of pesticide residues per sample

Thirty-nine percent (39%) of the total number of samples tested contained no residues at or above the reporting levels for each pesticide sought, with 25% of samples tested containing only 1 pesticide residue. Overall, this shows a low instance of residual pesticides across all samples tested over the 7-year sampling period.

6.8.5. Residue breakdown

Overall, 55 different pesticides were detected in the 2625 samples tested over the 7-year sampling period.

The 20 most frequently found residues have been tabulated in table 20 and Figure 26 (below) show these 20 most frequently found residues as a percentage of the total samples tested.

Table 20. Twenty most frequently detected pesticides, % frequency and concentration range (mg/kg)

Pesticide	Number of residues	Number of samples tested	Percentage of samples with residues / %	Residue range / mg/kg
chlormequat	671	849	79	0.010-14.4
glyphosate	442	1075	41	0.10-15
mepiquat	251	849	30	0.010-1.7
piperonyl butoxide	415	2219	19	0.010-6.0
tebuconazole	415	2219	19	0.010-0.36
deltamethrin	254	2397	11	0.010-0.72
fluxapyroxad	137	2219	6.2	0.010-0.19
azoxystrobin	96	2219	4.3	0.010-0.20
pirimiphos-methyl	92	2397	3.8	0.010-11
epoxiconazole	66	2219	3.0	0.010-0.13
chlorpyrifos-methyl	55	2397	2.3	0.010-0.46
cyprodinil	41	2219	1.8	0.011-0.39
cypermethrin	39	2397	1.6	0.012-0.55
pyraclostrobin	36	2219	1.6	0.010-0.044
fluroxypyr	38	2219	1.7	0.011-0.060
bixafen	33	2219	1.5	0.010-0.092
cyproconazole	28	2219	1.3	0.010-0.055
boscalid	27	2219	1.2	0.010-0.088
prothioconazole-desthio	21	2219	0.9	0.010-0.053
chlorpropham	18	2397	0.8	0.011-0.14

Another 35 pesticides with 8 or less residues were detected across the 2625 samples tested. The number of residues of these 35 pesticides amounts to 93 in total, with 17 of these 35 being single detections.

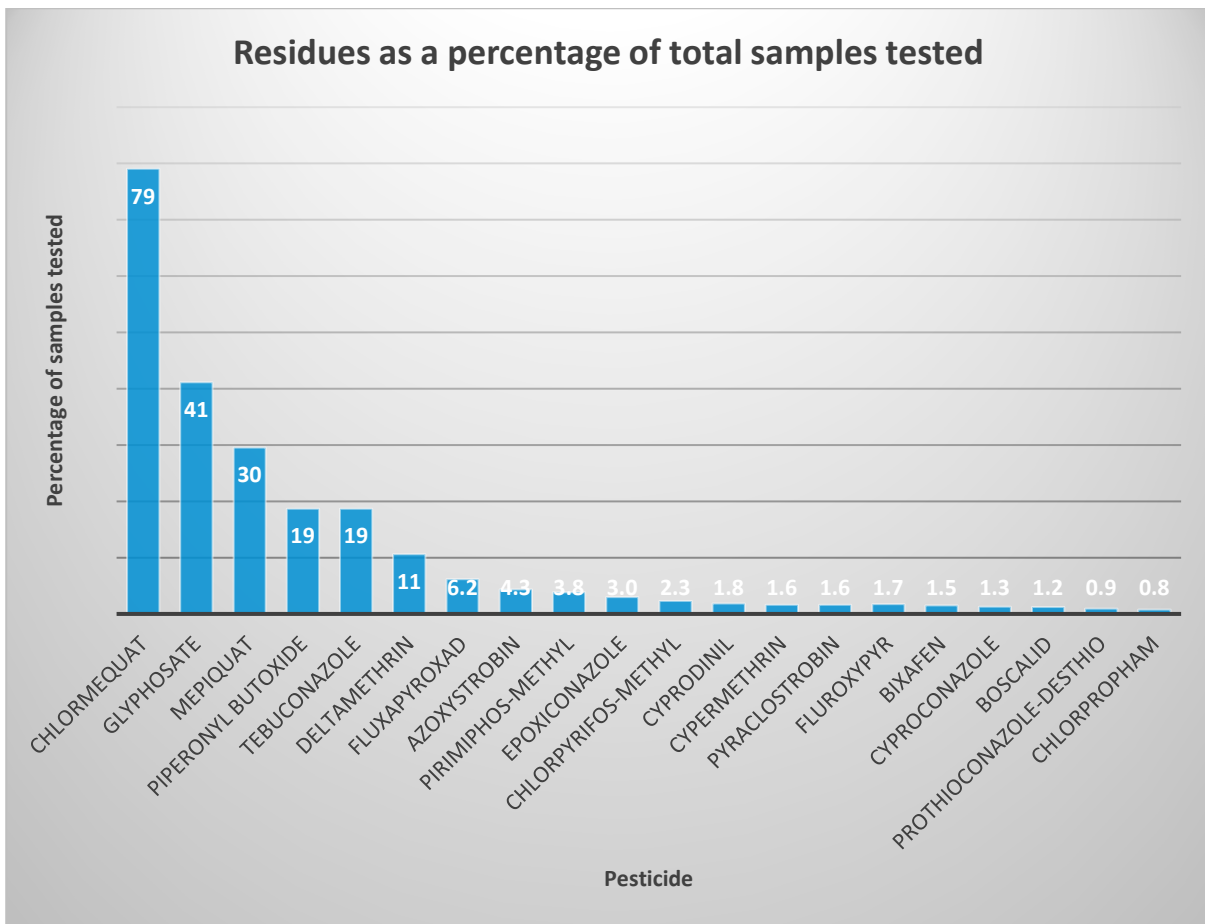


Figure 26. Twenty most frequently detected pesticides

As a percentage of total samples tested, plant growth regulators (chlormequat and mepiquat) were by far the most frequently found residues. Chlormequat was detected in 79% of all samples tested for plant growth regulators. The next most frequently detected pesticide residue was glyphosate, it was detected in 41% of samples tested for glyphosate.

6.8.6. MRL exceedances

Over the 7-year sampling period, 17 pesticide residues at or above their corresponding MRLs were detected in 16 samples. One sample of malt contained both 2-phenylphenol and biphenyl residues. Out of the 2625 samples tested, this equated to 0.6% of samples containing residues at or above their corresponding MRLs. These residues have been tabulated in table 21 below.

2-phenylphenol and biphenyl residues were from a peated malt sample and are thought to be contamination from the peating process and not from misuse.

Chlorpropham residues were thought to be from contaminated stores and not through application misuse. DDAC residues were thought to be from surface contact with disinfected equipment and not through misuse. All MRL exceedances are based on regulation 396/2005/EC [11].

Table 21. Summarised pesticide MRL exceedance results

Year	Pesticide	Commodity	Result / mg/kg	MRL / mg/kg
1	Chlorpyrifos	Food Oats	0.069	0.05
1	Chlorpropham	Malting Barley	0.047	0.01*
2	DDAC	Food Oats	0.17	0.1
3	DDAC	Milling Wheat	0.32	0.1
3	Chlorpropham	Milling Wheat	0.011	0.01*
3	Chlorpropham	Milling Wheat	0.019	0.01*
3	Chlorpropham	Milling Wheat	0.032	0.01*
3	Chlorpropham	Milling Wheat	0.011	0.01*
3	Chlorpropham	Food Oats	0.012	0.01*
4	Chlorpropham	Malting Barley	0.095	0.01*
4	Pirimiphos-methyl	Food Oats	10.3	5
5	2,4-DB	Malting Barley	0.067	0.05
6	Chlorpropham	Milling Wheat	0.032	0.01*
7	Chlorpropham	Milling Wheat	0.017	0.01*
7	Chlorpropham	Milling Wheat	0.028	0.01*
7	2-phenylphenol	Malt	0.079	0.02*
7	Biphenyl	Malt	0.180	0.01*

* Level at or about the limit of determination (LOD)

6.9. Additional analysis requests

Over the 7 years of the project, a range of additional analyses were carried out, this was usually in response to AHDB or trade partner requests, as well as recommendations raised as a result of horizon scanning. The requests were typically made before each year of analysis began and were specific to that year; therefore, no comparison data is available. The additional analysis requests were: Alternaria toxins, acrylamide, beauvericin + enniatins, aflatoxins, dioxins, PAHs, inorganic arsenic and sterigmatocystin.

6.9.1. Alternaria toxins

Five Alternaria toxins were analysed in malting barley and food oats. The only compounds detected in malting barley were alternariol and alternariol monomethyl ether. For food oats, only tenuazonic acid was detected.

Table 22. Summarised results of Alternaria toxins analysis

Alternaria Toxins (n=5) Harvest Results 2016							
	No. of Samples Analysed	% > LOD	Minimum Level µg/kg	Maximum Level µg/kg	Mean Level µg/kg	Mode Level µg/kg	Median Level µg/kg
Malting Barley ¹	40	10%	< 5	16	0.5	< 5	< 5
Food Oats ²	30	7%	0	84	3	< 10	< 10

¹ Malting Barley data is summary for alternariol and alternariol monomethyl ether, no other analytes detected.

² Food Oats data is summary for tenuazonic acid, no other analytes detected.

6.9.2. Acrylamide

During both years of acrylamide testing in malt samples, incidence levels were low (15%). The mean levels for both years were similar with the median levels being identical. However, whilst the overwhelming majority of samples analysed were below RL, in both years there were some samples where acrylamide was measured. In 2019, there were three samples (55, 104 and 1571 µg/kg) and in 2023 there were two (170 and 1521 µg/kg). Assimilated Regulation (EU) 2017/2158 sets maximum benchmark levels for acrylamide in various foodstuffs but does not include malt [12]. The levels of acrylamide measured are likely to be as a consequence of the kilning process.

Table 23. Summarised results of analysis of malt for acrylamide 2018–19

2018-2019	No. of Samples Analysed	% > LOD	Minimum Level µg/kg	Maximum Level µg/kg	Mean Level µg/kg	Median Level µg/kg
Malt	20	15	<30	1765	96.2	<30

Table 24. Summarised results of analysis of malt for acrylamide 2022–23

2022-2023	No. of Samples Analysed	% > Reporting Limit	Minimum Level µg/kg	Maximum Level µg/kg	Mean Level µg/kg	Median Level µg/kg
Malt	20	15	<30	1521	86.5	<30

6.9.3. Beauvericin + Enniatins

In 2019, 35 samples of milling wheats were tested for beauvericin and enniatins. There was a high incidence of beauvericin (91%), but the mean level measured was 4.1 µg/kg. Of the four enniatins, enniatin B1 was the most prevalent and had the highest maximum, mean and median levels. It should be noted that the RL of 1 µg/kg is very low, leading to the high incidence reporting. There are no maximum levels for these mycotoxins.

Table 25. Beauvericin and Enniatins results (µg/kg) for milling wheat samples in 2019

	No. of Samples Analysed	% > Reporting Limit	Minimum Level µg/kg	Maximum Level µg/kg	Mean Level µg/kg	Median Level µg/kg
Beauvericin	35	91%	<1	30.6	4.1	2.1
Enniatin A	35	26%	<1	8.5	<1	<1
Enniatin A1	35	69%	<1	44.7	4.9	2.6
Enniatin B	35	89%	<1	84.6	16.2	7.0
Enniatin B1	35	91%	<1	145	22.6	11.4

6.9.4. Sterigmatocystin

Food oats were analysed for sterigmatocystin in Year 1 (2017). Sterigmatocystin was detected in 37% of samples, with a range of less than RL, <0.2 µg/kg to 8.7 µg/kg. The mean level was 0.56 µg/kg, but the median value was <0.2 µg/kg.

Table 26. Sterigmatocystin results (µg/kg)

	No. of Samples Analysed	% > Reporting Limit	Minimum Level µg/kg	Maximum Level µg/kg	Mean Level µg/kg	Median Level µg/kg
Food Oats	30	37	<0.2	8.7	0.56	<0.2

6.9.5. Aflatoxins

Aflatoxins analyses were carried out in Year 6.

Ten matched pairs of malting barley and malt were analysed for aflatoxin with overall incidence being very low. Only AFB1 was detected, and the levels were all equal to the method RL. No ML exceedances were observed (Table 27).

Ten milling wheat samples were also analysed for aflatoxins. The only residue detected was 0.2 µg/kg AFB1, just above the RL, in one sample of milling wheat from Germany.

Table 27. Aflatoxins results ($\mu\text{g}/\text{kg}$) in matched pairs of malting barley and malt

	AFB1	AFB2	AFG1	AFG2	total
Malting Barley					
No. of Samples	10	10	10	10	10
% > LOD	10%	0%	0%	0%	10%
Minimum Level $\mu\text{g}/\text{kg}$	<0.2	<0.2	<0.2	<0.2	<0.8
Maximum Level $\mu\text{g}/\text{kg}$	0.2	<0.2	<0.2	<0.2	0.2
Mean Level $\mu\text{g}/\text{kg}$	<0.2	<0.2	<0.2	<0.2	<0.8
Median Level $\mu\text{g}/\text{kg}$	<0.2	<0.2	<0.2	<0.2	<0.8
Malt					
No. of Samples	10	10	10	10	10
% > LOD	40%	0%	0%	0%	40%
Minimum Level $\mu\text{g}/\text{kg}$	<0.2	<0.2	<0.2	<0.2	<0.8
Maximum Level $\mu\text{g}/\text{kg}$	0.2	<0.2	<0.2	<0.2	0.2
Mean Level $\mu\text{g}/\text{kg}$	0.1	<0.2	<0.2	<0.2	0.1
Median Level $\mu\text{g}/\text{kg}$	<0.2	<0.2	<0.2	<0.2	<0.8

6.9.6. Dioxin-like and non-dioxin-like PCBs

In Year 1, feed samples were analysed for dioxin-like (DL) and non-dioxin-like PCBs (ICES-6). All concentrations measured were found to be low and below any regulated limits (where applicable) (Table 28).

Table 28. Results of dioxins analyses of feed products, Year 1 2016–17

	No. of samples analysed		Min/Max Range Upper bound TEQ (ng/kg) as received
Feed Barley	29	Dioxins and furans	0.03 - 0.03
		DL-PCBs	0.02 - 0.02
		Sum Dioxins/furans/DL-PCBS	0.05 - 0.05
		Sum of ICES-6 (µg/kg)	0.06 - 0.15
Feed Wheat	40	Dioxins and furans	0.03 - 0.03
		DL-PCBs	0.02 - 0.02
		Sum Dioxins/furans/DL-PCBS	0.05 - 0.05
		Sum of ICES-6 (µg/kg)	0.06 - 0.07
Feed Oats	10	Dioxins and furans	0.03 - 0.03
		DL-PCBs	0.02 - 0.02
		Sum Dioxins/furans/DL-PCBS	0.05 - 0.05
		Sum of ICES-6 (µg/kg)	0.06 - 0.06

6.9.7. PAHs

Feed samples were also analysed for PAHs in Year 1. All feed concentrations were found to be low, there are no regulated maximum levels for feed.

Table 29. Results of PAHs analyses in feed products, Year 1 2016–17

	No. of samples analysed		Min/Max Range Upper bound (µg/kg) as received
Feed Barley	29	Sum of PAH 4	0.13 - 7.5
		benzo(a)pyrene	<0.04 - 1.83
Feed Wheat	40	Sum of PAH 4	0.12 - 2.87
		benzo(a)pyrene	<0.04 - 0.58
Feed Oats	10	Sum of PAH 4	0.63 - 8.06 ²
		benzo(a)pyrene	0.13 - 2.59 ¹
¹ Indicative value			
² Includes indicative value from ¹ above			

7. Dissemination activities

Throughout the project, the results have been summarised each year in an Annual Report and shared on the project website at:

<https://ahdb.org.uk/monitoring-of-contaminants-in-uk-cereals-used-for-processing-food-and-animal-feed>

In addition, results are shared by the partners among their member companies.

All contaminants results from this project have been formatted into the required format and submitted to EFSA to be included in their data sets.

Two articles describing the project were published. An article in Crop Production Magazine, June 2018 highlighted the issue of ergot alkaloids and reported some results from the project.

<https://cereals.ahdb.org.uk/media/1397450/T2F-June-2018-Ergot-alkaloids-under-the-spotlight.pdf>.

A second article about the project was published in Arable Farming in June 2020

<https://www.farmersguardian.com/feature/4090835/arable-farming-magazines-june-2020-digital-edition>.

Several presentations about the project have been given, including at UK Stakeholder events for mycotoxins in November 2021 and December 2022 with attendance from FSA and industry.

A presentation titled 'Ergot Alkaloids in Cereals, Results from industry monitoring – a UK perspective' was accepted for presentation at the World Mycotoxin Forum, Ghent, Belgium in October 2023.

8. Discussion

In general, the data gathered from this project indicates that the majority of cereals grown in the UK adhere to both EU and UK laws and guidelines concerning the presence of contaminants.

Mycotoxins: Full descriptions are given in Section 5 of the report.

DON – Was routinely measured in all food and feed products throughout the project.

Concentrations of the vast majority of samples surveyed were well below maximum permitted levels. DON was the most frequently detected trichothecene throughout the 7-year project.

T-2 and HT-2 – Oat products had the highest detection rate throughout the project, with occurrence routinely at 100%, with 2023 being the lowest (70% - food oats). As stated in 5.2.1, there were several occasions when T-2 + HT-2 levels were detected above the indicative levels set by Commission Recommendation 2013/165/EU [9]. Although, most results were below the

Indicative levels, it is important to note that this is a changing landscape. Recently Commission Regulation (EU) 2024/1038 was published [13]. This introduced maximum levels for T-2 and HT-2 toxins in the EU from 1 July 2024. These new MLs are set much lower than the previous levels in the Recommendation 2013/165/EU [9]. Several maximum levels reported for food oats, milling wheat and malting barley throughout this project would exceed the new EU levels. It is important to note these MLs do not apply in Great Britain. The Food Standards Agency published a call for T-2 and HT-2 toxin data in 2023. All data from this project was submitted in response to this call. All submitted data will be used by the FSA to carry out their own risk assessment on T-2 and HT-2 toxins and support decisions about what, if any, risk management measures should be taken in GB.

Ergot Alkaloids – Incidence levels were generally high for ergot alkaloids, with ergot alkaloids measured in most products at greater than 50% each year. Although incidence levels were high, the actual concentrations found were generally low. Some ‘high’ values were measured throughout the project; however, this was still less than 1% all samples measured. Industry have noted there is still an issue whereby visible sclerotia have been removed from samples, yet the chemical analysis still results in ‘high’ values of ergot alkaloids being measured. It is, therefore, essential that this monitoring and analysis continues, and an even greater emphasis placed on ensuring a representative sample is taken.

Ochratoxin A – Less frequently detected in food grains, and concentrations were generally very low, with mean values generally below the reporting limit (0.2 µg/kg). Throughout the project, only 4 samples exceeded MLs (3 food oats and 1 milling wheat). This suggests that toxin synthesis in food grains is effectively controlled by good practice during storage conditions. The incidence in compounded feed samples was similar to that observed in the previous study, with wheatfeed and oatfeed concentrations significantly higher but still well below guideline levels.

Pesticides – More descriptions are given in Section 5.8 of the report.

- Plant growth regulators were the most commonly detected residues and were consistent year on year
- Fungicides were commonly detected but also showed the greatest year-on-year differences. This may have been due to seasonal weather patterns
- Glyphosate was commonly detected and did show year-to-year variance. This was likely due to seasonal weather patterns
- Insecticides were commonly detected and over the last 2 years of the survey, a noticeable decrease in their average residues per sample has been observed
- Piperonyl butoxide was commonly found and shows little variance in its detection

- Extra non-core compounds detected have been steady with little variance. Although, Year 7 (Harvest Year 2022) showed almost double the average residue per sample compared to previous years. This may be due to seasonal weather patterns
- Chlorpropham had few detections but was consistently detected each year over the 7-year period with no characteristic pattern. Its occurrence was thought to be a result of contamination from contaminated former potato stores where it can reside for many years in the building fabric.

Metals – Section 5.6 focuses on milling wheat and food oats; however, all products were analysed for metals at different points throughout the project. Concentrations of heavy metals were generally low in the samples tested and well below current legal limits, this also aligns with measurements taken during the previous 4-year study. As per the previous study, the nickel monitoring requested by EFSA has continued (along with aluminium and copper) and although incidence was high, the concentrations measured were low.

Considerations and suggestions for future years – Sampling for mycotoxins is inherently difficult due to the heterogenous distribution of possible mycotoxins contamination. This can lead to difficulties with obtaining representative samples. For this project and for most partners, 1 kg samples are submitted to the laboratory, this sample is taken from several tonnes. While this sample size does not fully meet the requirements of Assimilated Commission Regulation (EC) No 401/2006 [14] several things should be borne in mind. The sampling regulation is designed to be used for monitoring for enforcement and regulatory purposes, this study is intended to obtain an ongoing snapshot of the occurrence of mycotoxins and other contaminants in cereals and is not intended to be used for official purposes. Using the sampling protocol in the regulation would result in much larger samples size which would substantially increase the costs incurred to collect, transport and homogenise the sample before analysis, as well as adding significantly to the time required to do this. The sample size used in this project is a reasonable compromise as it is sufficiently large. Any positive findings (ML exceedances) are reported immediately to partners to take follow up investigative action which may involve further sampling and analysis.

A further consideration is the changing landscape for Regulations. There are already several examples of divergence between Great Britain and the European Union. Commission Regulation (EU) 2023/915 [15] bringing together all the amendments to the Regulation EC (No) 1881/2006 [7] was published in 2023, this fully replaced Regulation 1881/2006 in the EU which remains in force in GB. Further changes that are due to come into force later in 2024 that will apply in the EU have already been published. There is a further complication that EU MLs apply in Northern Ireland and some samples for this study originate from there. Some consideration will need to be made for any future work on how to deal with these different MLs.

9. Acknowledgments

We would like to express our deepest thanks to the following colleagues, trade body partners and additional support staff who contributed to the successful delivery of this project.

AHDB

Dhan Bandari

UK Flour Millers

Joe Brennan

UK Flour Millers consider this project to be the largest single source of UK grain contaminant data, covering the primary outputs of UK arable farms. It provides an understanding of absolute contamination, as well as seasonal variation in contaminants affecting wheat, barley and oats. Crucially, the project provides a source of independent data and covers multiple contaminants in the same samples, making it relatively unique in the context of contaminant monitoring. Additionally, the project facilitates a forum within which the UK cereal processing sectors can meet and discuss contaminant data and mitigation for the supply chain. The project also provides evidence that can be used in relation to EU limits, such as ergot alkaloids, and cereal contaminant policymaking in general. In doing so, contaminant legal limits are set in a more practical manner, delivering a significant saving for the arable chain.

AIC

Edwin Snow

Rose Riby

James McCulloch

AIC statement: Since 2016, this project has monitored residues and contaminants in cereals, providing the feed and combinable crop industry with excellent data. It helps to inform strategic decisions in cereal marketing and feed-material sourcing. It also helps confirm the industry's reputation for compliance and quality, ensuring that grain produced by UK farmers meets regulatory requirements and can freely enter the food, feed and fuel supply chains. It also contributes concrete evidence of contaminant burdens, which is passed on to regulators (in the UK and EU) and plays a vital role in informing pragmatic policy decisions. Given the prospect of new EU legislation on mycotoxin levels in food and feed materials and finished feed, it is more important than ever for the industry to have accurate data on a wide range of food and feed material contaminants. By satisfying the requirements of EU legislation, it allows the UK to maintain access to this valuable market for our domestic food and feed sectors.

MAGB

Julian South

Sue Capewell

The MAGB consider the AHDB Contaminants project to be an essential part of the overall due diligence for malting barley growers and the malting industry in the UK. The results concur well with the MAGB's own programme of testing and that undertaken by their member companies. The combination of these analytical programmes ensures that all contaminants are included in sufficient numbers and in proportion to the risks to product safety. Additional benefits of the programme are the horizon scanning activity and the contribution to the discussion around new regulatory limits at UK and EU levels. The high level of expertise engaged ensures that the best analytical technology is used and identifies any gaps requiring further research under the AHDB programme. This is also one of the few schemes that allows comparisons of contaminants levels across cereal types (barley, wheat, oats etc.) as well as by end user category (e.g. food vs feed).

BOBMA

Indika Pathirathna

Derek Croucher

From BOBMA's perspective, the AHDB Contaminants Monitoring Project is critical for the UK cereal processing industries. The outputs are the largest source of independent validated data on cereal contaminant levels demonstrating ongoing compliance with UK (and where relevant, EU) Regulations. As the project covers multiple contaminants in the same samples, it provides the industry a unique view in the context of contaminant monitoring. These results not only provide an understanding of the actual contamination levels but also other factors affecting contaminants in UK grown cereals and considers emerging risks. Furthermore, the project facilitates a forum within which the UK cereal processing sectors can meet and discuss contaminant data, upcoming challenges, and mitigation for the supply chain. It is these aspects of the project that deliver significant value above and beyond the direct value of the monitoring data itself. The relevance of these factors can be demonstrated by how the project has supported oat and barley sectors over the years when engaging with both European and UK regulators in relation to setting maximum limits for cereal contaminants such as T-2/HT-2, nickel and ergot alkaloids. In doing so, contaminant legal limits are set in a more practical manner, delivering a significant benefit for the arable chain.

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12. Commission Regulation (EU) 2017/2158 of 20 November 2017 establishing mitigation measures and benchmark levels for the reduction of the presence of acrylamide in food <https://www.legislation.gov.uk/eur/2017/2158/contents>
13. Commission Regulation (EU) 2024/1038 of 9 April 2024 amending Regulation (EU) 2023/915 as regards maximum levels of T-2 and HT-2 toxins in food https://eur-lex.europa.eu/legal-content/EN/TXT/?uri=OJ:L_202401038
14. Commission Regulation (EC) No 401/2006 of 23 February 2006 laying down the methods of sampling and analysis for the official control of the levels of mycotoxins in foodstuffs <https://www.legislation.gov.uk/eur/2006/401/data.pdf>
15. Commission Regulation (EU) 2023/915 of 25 April 2023 on maximum levels for certain contaminants in food and repealing Regulation (EC) No 1881/2006 (Text with EEA relevance) <https://eur-lex.europa.eu/legal-content/EN/TXT/?uri=CELEX%3A32023R0915>

11. Appendices

11.1. Appendix 1. Table of Pesticides Reporting Limits.

Pesticide	RL / mg/kg	Pesticide	RL / mg/kg
2,4-D	<0.01	bromuconazole	<0.01
2,4-DB	<0.01	bupirimate	<0.01
2-phenylphenol	<0.02	buprofezin	<0.01
6-benzyl aminopurine	<0.01	butachlor	<0.01
abamectin	<0.01	butocarboxim	<0.01
acephate	<0.01	butocarboxim sulfoxide	<0.01
acetamiprid	<0.01	butoxycarboxim	<0.01
acetochlor	<0.01	cadusafos	<0.01
acibenzolar-S-methyl	<0.01	carbaryl	<0.01
aclonifen	<0.01	carbendazim	<0.01
acrinathrin	<0.01	carbetamide	<0.01
alachlor	<0.01	carbofuran	<0.001
aldicarb	<0.01	carbofuran (3-hydroxy)	<0.001
aldicarb sulfone	<0.01	carboxin	<0.01
aldicarb sulfoxide	<0.01	chlorantraniliprole	<0.01
aldrin	<0.01	chlorbufam	<0.01
allethrin	<0.01	chlordane (cis)	<0.01
ametoctradin	<0.01	chlordane (trans)	<0.01
amidosulfuron	<0.01	chlorfenapyr	<0.01
asulam	<0.01	chlorfenvinphos	<0.01
atrazine	<0.01	chlorfluazuron	<0.01
azinphos-ethyl	<0.01	chloridazon	<0.01
azinphos-methyl	<0.01	chlorobenzilate	<0.01
azoxystrobin	<0.01	chlorothalonil	<0.01
BAC10	<0.05	chlorpropham	<0.01
BAC12	<0.05	chlorpyrifos	<0.01
BAC14	<0.05	chlorpyrifos-methyl	<0.01
BAC16	<0.05	chlorthal-dimethyl	<0.01
benalaxyl	<0.01	chlortoluron	<0.01
bendiocarb	<0.01	chlozolinate	<0.01
benthiavalicarb-isopropyl	<0.01	chromafenozide	<0.01
bifenox	<0.01	clethodim	<0.01
bifenthrin	<0.01	clofentezine	<0.01
biphenyl	<0.01	clomazone	<0.01
bispyribac-sodium	<0.01	clothianidin	<0.01
bitertanol	<0.01	coumaphos	<0.01
bixafen	<0.01	cyanazine	<0.01
boscalid	<0.05	cyantraniliprole	<0.01
bromophos-ethyl	<0.01	cyazofamid	<0.01
bromopropylate	<0.01	cycloate	<0.01
bromoxynil	<0.01	cycloxydim	<0.01

Pesticide	RL / mg/kg	Pesticide	RL / mg/kg
cyflufenamid	<0.01	disulfoton	<0.01
cyfluthrin	<0.01	disulfoton sulfone	<0.01
cyhalofop butyl	<0.01	diuron	<0.01
cyhalothrin-lambda	<0.01	DMF	<0.01
cymoxanil	<0.01	DMPF	<0.01
cypermethrin	<0.01	DMSA	<0.01
cyproconazole	<0.01	dodine	<0.01
cyprodinil	<0.01	emamectin benzoate	<0.01
cyromazine	<0.01	endosulfan (I)	<0.01
DDAC	<0.05	endosulfan (II)	<0.01
DDD-pp	<0.01	endosulfan sulfate	<0.01
DDE-pp	<0.01	endrin	<0.01
DDT-op	<0.01	EPN	<0.01
DDT-pp	<0.01	epoxiconazole	<0.01
deltamethrin	<0.01	EPTC	<0.01
demeton-S-methyl	<0.01	ethiofencarb	<0.01
demeton-S-methyl sulfone	<0.01	ethiofencarb sulfone	<0.01
desmedipham	<0.01	ethiofencarb sulfoxide	<0.01
diafenthiuron	<0.01	ethion	<0.01
diazinon	<0.01	ethiprole	<0.01
dichlobenil	<0.01	ethirimol	<0.01
dichlofluanid	<0.01	ethofumesate	<0.01
dichlorprop	<0.01	ethoprophos	<0.01
dichlorvos	<0.01	etofenprox	<0.01
diclobutrazol	<0.01	etoxazole	<0.01
dicloran	<0.01	etridiazole	<0.01
dicofol	<0.01	etrimfos	<0.01
dicrotophos	<0.01	famoxadone	<0.01
dieldrin	<0.01	fenamidone	<0.01
diethofencarb	<0.01	fenamiphos	<0.01
difenoconazole	<0.01	fenamiphos sulfone	<0.01
diflubenzuron	<0.01	fenamiphos sulfoxide	<0.01
diflufenican	<0.01	fenarimol	<0.01
dimethenamid	<0.01	fenazaquin	<0.01
dimethoate	<0.01	fenbuconazole	<0.01
dimethomorph	<0.01	fenbutatin oxide	<0.01
dimoxystrobin	<0.01	fenhexamid	<0.01
diniconazole	<0.01	fenitrothion	<0.01
dinoseb	<0.01	fenoprop	<0.01
dinotefuran	<0.01	fenoxycarb	<0.01
diphenylamine	<0.05	fenpropathrin	<0.01

Pesticide	RL / mg/kg	Pesticide	RL / mg/kg
fenpropidin	<0.01	furathiocarb	<0.001
fenpropimorph	<0.01	halofenozide	<0.01
fenpyrazamine	<0.01	halosulfuron-methyl	<0.01
fenpyroximate	<0.01	haloxyfop (free acid)	<0.01
fensulfothion	<0.01	HCH-alpha	<0.01
fensulfothion sulfone	<0.01	HCH-beta	<0.01
fensulfothion-oxon	<0.01	HCH-gamma	<0.01
fensulfothion-oxon-sulfone	<0.01	heptachlor	<0.01
fenthion	<0.01	heptachlor epoxide-cis	<0.01
fenthion sulfone	<0.01	heptachlor epoxide-trans	<0.01
fenthion sulfoxide	<0.01	heptenophos	<0.01
fentin acetate	<0.05	hexachlorobenzene	<0.01
fenvalerate	<0.01	hexaconazole	<0.01
fipronil	<0.002	hexazinone	<0.01
fipronil de-sulfinyl	<0.002	hexythiazox	<0.01
fipronil sulfone	<0.002	imazalil	<0.01
flonicamid	<0.01	imazaquin	<0.01
fluazifop (free acid)	<0.01	imidacloprid	<0.01
fluazifop-p-butyl	<0.01	indoxacarb	<0.01
fluazinam	<0.01	ioxynil	<0.01
flubendiamide	<0.01	iprodione	<0.01
flucythrinate	<0.01	iprovalicarb	<0.01
fludioxonil	<0.01	isazofos	<0.01
flufenacet	<0.01	isocarbofos	<0.01
flufenoxuron	<0.01	isofenphos	<0.01
fluometuron	<0.01	isofenphos-methyl	<0.01
fluopicolide	<0.01	isoprocarb	<0.01
fluopyram	<0.01	isoprothiolane	<0.01
fluxastrobin	<0.01	isoproturon	<0.01
fluquinconazole	<0.01	isopyrazam	<0.01
flurochloridone	<0.01	isoxaben	<0.01
fluroxypyr	<0.01	isoxaflutole	<0.01
flusilazole	<0.01	kresoxim-methyl	<0.01
flutolanil	<0.01	lenacil	<0.01
flutriafol	<0.01	linuron	<0.01
fluvalinate	<0.01	lufenuron	<0.01
fluxapyroxad	<0.01	malaoxon	<0.01
fonofos	<0.01	malathion	<0.01
formetanate-HCl	<0.01	mandipropamid	<0.01
fosthiazate	<0.01	MCPA	<0.01
furalaxyl	<0.01	MCPB	<0.01

Pesticide	RL / mg/kg	Pesticide	RL / mg/kg
mecarbam	<0.01	ofurace	<0.01
mecoprop	<0.01	omethoate	<0.01
mepanipyrim	<0.01	oxadiargyl	<0.01
mephosfolan	<0.01	oxadiazon	<0.01
mepronil	<0.01	oxadixyl	<0.01
mesosulfuron-methyl	<0.01	oxamyl	<0.01
metaflumizone	<0.01	oxasulfuron	<0.01
metalaxyl	<0.01	oxychlorane	<0.01
metamitron	<0.01	oxydemeton-methyl	<0.01
metazachlor	<0.01	oxyfluorfen	<0.01
metconazole	<0.01	paclobutrazol	<0.01
methabenzthiazuron	<0.01	paraoxon-methyl	<0.01
methacrifos	<0.01	parathion-ethyl	<0.01
methamidophos	<0.01	parathion-methyl	<0.01
methidathion	<0.01	penconazole	<0.01
methiocarb	<0.01	pencycuron	<0.01
methiocarb sulfone	<0.01	pendimethalin	<0.02
methiocarb sulfoxide	<0.01	penflufen	<0.01
methomyl	<0.01	pentachloroaniline	<0.01
methoxychlor	<0.01	pentanochlor	<0.01
methoxyfenozide	<0.01	penthiopyrad	<0.01
metobromuron	<0.01	permethrin	<0.01
metolachlor	<0.01	phenmedipham	<0.01
metolcarb	<0.01	phenthoate	<0.01
metosulam	<0.01	phorate	<0.01
metoxuron	<0.01	phorate sulfone	<0.01
metrafenone	<0.01	phorate sulfoxide	<0.01
metribuzin	<0.01	phosalone	<0.01
metsulfuron-methyl	<0.01	phosmet	<0.01
mevinphos	<0.01	phosphamidon	<0.01
molinate	<0.01	phoxim	<0.01
monocrotophos	<0.01	phthalimide	<0.01
monolinuron	<0.01	picloram	<0.01
monuron	<0.01	picolinafen	<0.01
myclobutanil	<0.01	picoxystrobin	<0.01
napropamide	<0.01	piperonyl butoxide	<0.01
nitenpyram	<0.01	pirimicarb	<0.01
nitrofen	<0.01	pirimicarb-desmethyl	<0.01
nitrothal-isopropyl	<0.01	pirimiphos-ethyl	<0.01
novaluron	<0.01	pirimiphos-methyl	<0.01
nuarimol	<0.01	prochloraz	<0.01

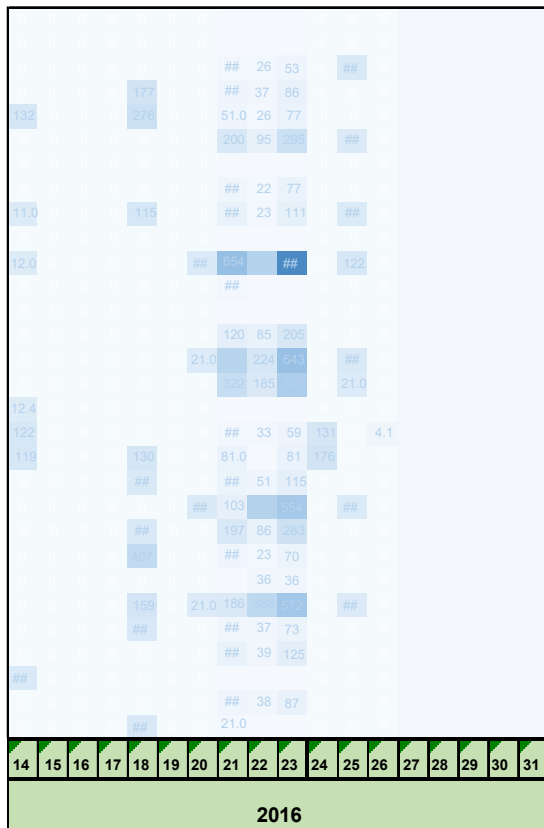
Pesticide	RL / mg/kg	Pesticide	RL / mg/kg
procymidone	<0.01	spirodiclofen	<0.01
profenofos	<0.01	spiromesifen	<0.01
promecarb	<0.01	spirotetramat	<0.01
prometryn	<0.01	spirotetramat enol	<0.01
propachlor	<0.01	spiroxamine	<0.01
propamocarb (free base)	<0.01	sulcotrione	<0.01
propaquizafop	<0.01	sulfoxaflor	<0.01
propargite	<0.01	tebuconazole	<0.01
propetamphos	<0.01	tebufenozide	<0.01
propham	<0.01	tebufenpyrad	<0.05
propiconazole	<0.01	tebupirimphos	<0.01
propoxur	<0.01	tebuthiuron	<0.01
propyzamide	<0.01	tecnazene	<0.01
proquinazid	<0.01	teflubenzuron	<0.01
prosulfocarb	<0.01	tefluthrin	<0.01
prosulfuron	<0.01	tepraloxydim	<0.01
prothioconazole-desthio	<0.01	terbufos	<0.01
prothiofos	<0.01	terbufos sulfone	<0.01
pymetrozine	<0.01	terbufos sulfoxide	<0.01
pyraclostrobin	<0.01	terbuthylazine	<0.01
pyrazophos	<0.01	terbutryn	<0.01
pyrethrins	<0.01	tetrachlorvinphos	<0.01
pyridaben	<0.01	tetraconazole	<0.01
pyridalyl	<0.01	tetradifon	<0.01
pyridaphenthion	<0.01	tetrahydrophthalimide	<0.01
pyrifenox	<0.01	tetramethrin	<0.05
pyrimethanil	<0.01	TFNA	<0.01
pyriproxyfen	<0.01	TFNG	<0.01
quassia	<0.01	thiabendazole	<0.01
quinalphos	<0.01	thiacloprid	<0.01
quinmerac	<0.01	thiamethoxam	<0.01
quinoclamine	<0.01	thiodicarb	<0.01
quinoxifen	<0.01	thiophanate-methyl	<0.01
quintozene	<0.01	tolclofos-methyl	<0.01
quizalofop P	<0.01	tolfenpyrad	<0.01
rimsulfuron	<0.01	tolyfluanid	<0.01
rotenone	<0.01	triadimefon	<0.01
simazine	<0.01	triadimenol	<0.01
spinetoram	<0.01	triallate	<0.01
spinetroram	<0.01	triasulfuron	<0.01
spinosad	<0.01	triazamate (free acid)	<0.01

Pesticide	RL / mg/kg	Pesticide	RL / mg/kg
triazophos	<0.01	trifluralin	<0.01
triclopyr	<0.01	triforine	<0.01
tricyclazole	<0.01	triticonazole	<0.01
trifloxystrobin	<0.01	vinclozolin	<0.01
triflumizole	<0.01	zoxamide	<0.01
triflumuron	<0.01		

11.2. Appendix 2. Co-occurrence tables

For all charts below, the specific analytes run along the x axis with the samples increasing (from sample 1) upwards along the y axis. The purpose of the charts is to display a snapshot of the mycotoxin profile of the product type submitted for a given year. Therefore, extracting specific sample numbers or values is not expected. For more information regarding specific results, please refer to section 6.

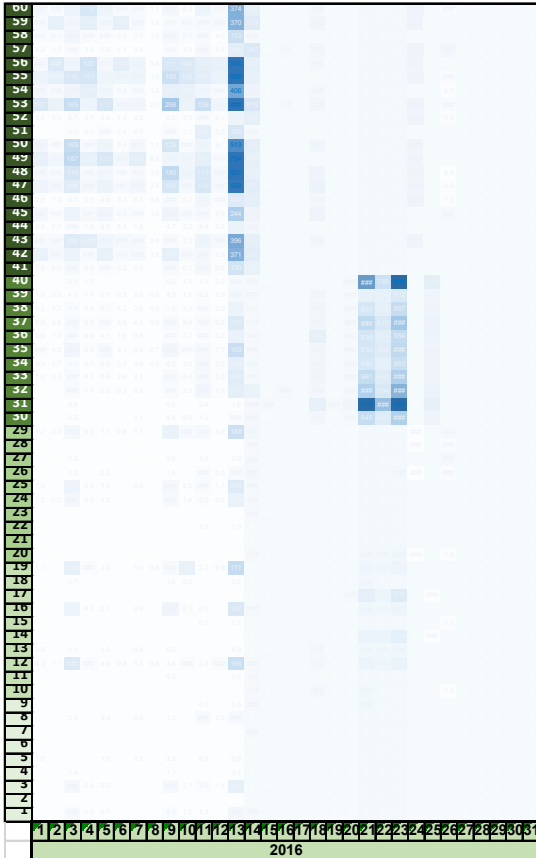
11.2.1. BOBMA



1. Ergocornine
2. Ergocorninine
3. Ergocristine
4. Ergocristinine
5. a+b-Ergocryptine
6. a+b-Ergocryptinine
7. Ergometrine
8. Ergometrinine
9. Ergosine
10. Ergosinine
11. Ergotamine
12. Ergotaminine
13. Total Ergots (upper bound)

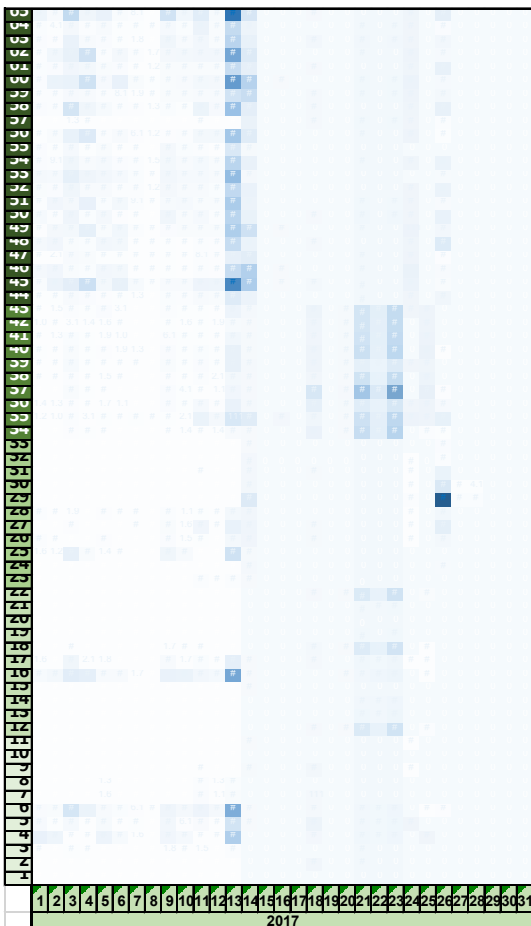
14. DON
15. FUS-X
16. 3Ac DON
17. 15Ac DON
18. NIV
19. DAS
20. NEO
21. HT-2
22. T-2
23. HT-2 + T-2
24. DON-3-Glc
25. T-2- α 3-Glc
26. ZEN
27. α -ZEL
28. β -ZEL
29. ZEN-14-Glc
30. α -ZEL-14-Glc
31. β -ZEL-14-Glc

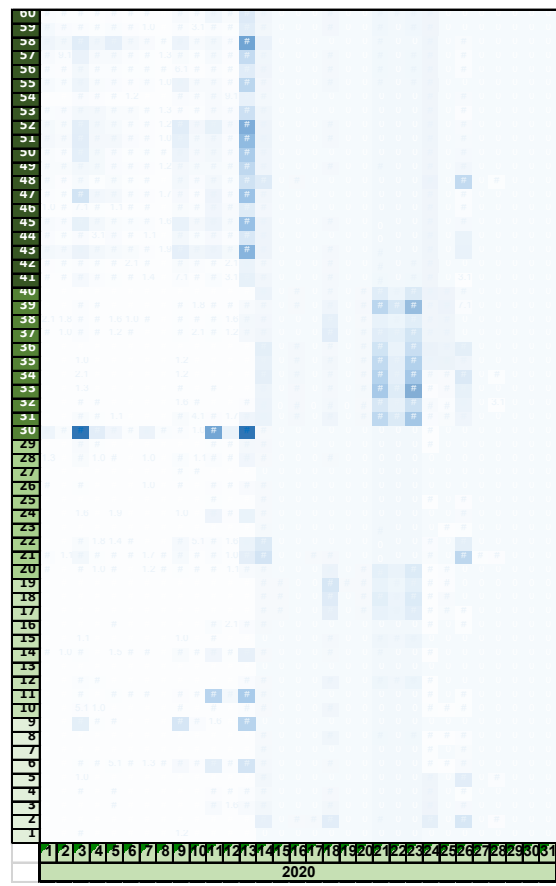
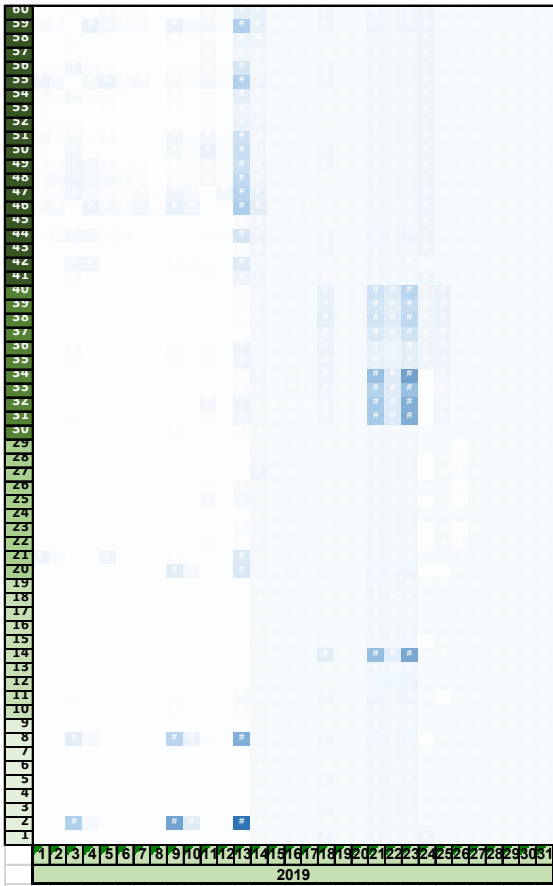
11.2.2. AIC



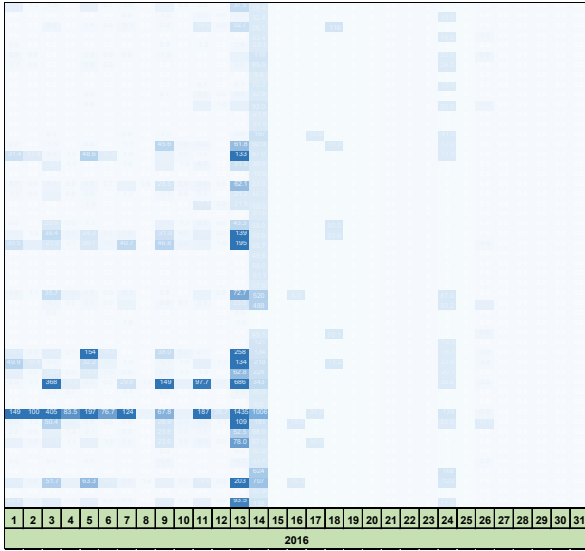
Key	
Sample No.	Sample Type
1 - 9	Feed Barley
10 - 19	Feed Oats
20 - 29	Feed Wheat
30 - 40	Oatfeed
41 - 60	Wheatfeed

- | | |
|--------------------------------|--------------------------|
| 1. Ergocornine | 14. DON |
| 2. Ergocorninine | 15. FUS-X |
| 3. Ergocristine | 16. 3Ac DON |
| 4. Ergocristinine | 17. 15Ac DON |
| 5. a+b-Ergocryptine | 18. NIV |
| 6. a+b-Ergocryptinine | 19. DAS |
| 7. Ergometrine | 20. NEO |
| 8. Ergometrinine | 21. HT-2 |
| 9. Ergosine | 22. T-2 |
| 10. Ergosinine | 23. HT-2 + T-2 |
| 11. Ergotamine | 24. DON-3-Glc |
| 12. Ergotaminine | 25. T-2- α 3-Glc |
| 13. Total Ergots (upper bound) | 26. ZEN |
| | 27. α -ZEL |
| | 28. β -ZEL |
| | 29. ZEN-14-Glc |
| | 30. α -ZEL-14-Glc |
| | 31. β -ZEL-14-Glc |



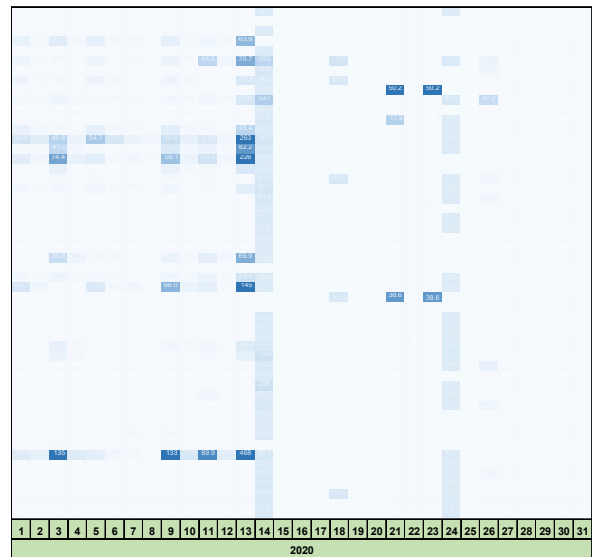
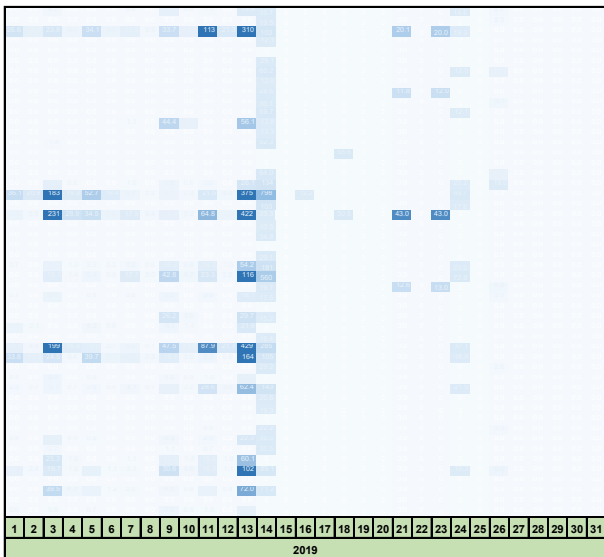
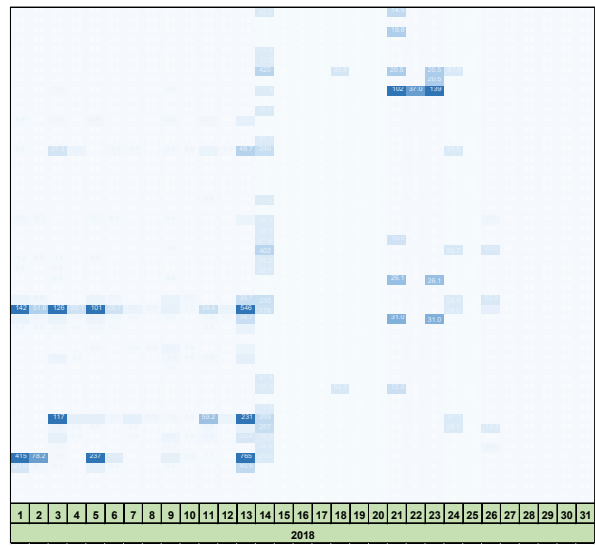


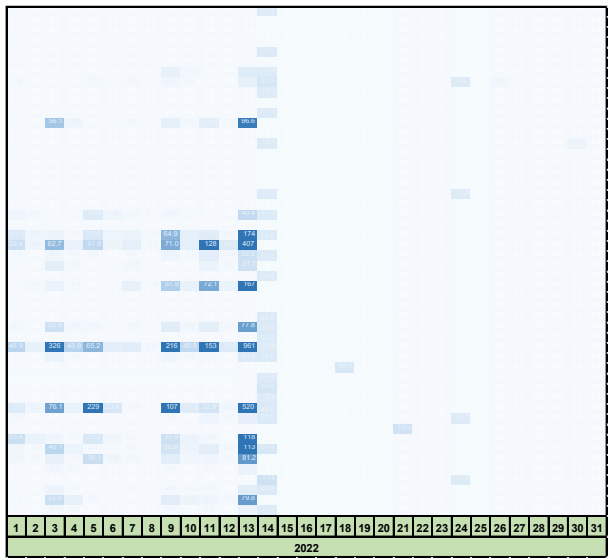
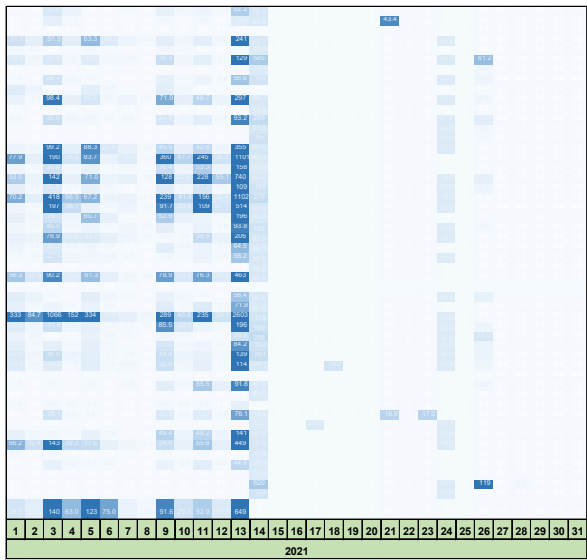
11.2.3. UKFM



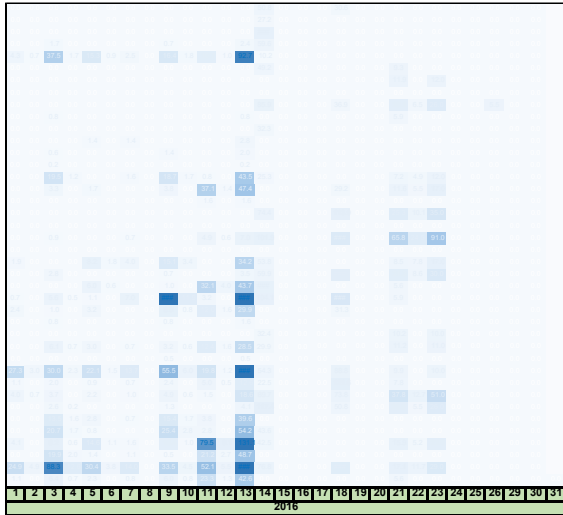
1. Ergocornine
2. Ergocorninine
3. Ergocristine
4. Ergocristinine
5. a+b-Ergocryptine
6. a+b-Ergocryptinine
7. Ergometrine
8. Ergometrinine
9. Ergosine
10. Ergosinine
11. Ergotamine
12. Ergotaminine
13. Total Ergots (upper bound)

14. DON
15. FUS-X
16. 3Ac DON
17. 15Ac DON
18. NIV
19. DAS
20. NEO
21. HT-2
22. T-2
23. HT-2 + T-2
24. DON-3-Glc
25. T-2- α 3-Glc
26. ZEN
27. α -ZEL
28. β -ZEL
29. ZEN-14-Glc
30. α -ZEL-14-Glc
31. β -ZEL-14-Glc





11.2.4. MAGB



1. Ergocornine
2. Ergocorninine
3. Ergocristine
4. Ergocristinine
5. a+b-Ergocryptine
6. a+b-Ergocryptinine
7. Ergometrine
8. Ergometrinine
9. Ergosine
10. Ergosinine
11. Ergotamine
12. Ergotaminine
13. Total Ergots (upper bound)

14. DON
15. FUS-X
16. 3Ac DON
17. 15Ac DON
18. NIV
19. DAS
20. NEO
21. HT-2
22. T-2
23. HT-2 + T-2
24. DON-3-Glc
25. T-2- α 3-Glc
26. ZEN
27. α -ZEL
28. β -ZEL
29. ZEN-14-Glc
30. α -ZEL-14-Glc
31. β -ZEL-14-Glc

