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RD-2012-3779 Monitoring of mycotoxins and other contaminants in UK cereals used in malting, milling & animal feed.

by

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1. Abstract

This project looked at the occurrence of key contaminants in UK-grown cereals in UK-grown wheat, barley and oats and their co-products. The project covered samples from the 2012, 2013, 2014 and 2015 harvests intended for use in the milling, malting and animal feed industries. Samples of each type of grain were collected immediately after harvest and after storage of up to six months. Relevant contaminants were selected and a sampling programme agreed by a steering group comprising AHDB, the Trade Associations nabim, MAGB, and AIC. Campden BRI informed the steering group through “horizon scanning” of official publications and scientific and agricultural literature as well as advice from scientists from the analytical laboratories. The contaminants selected were pesticides, growth regulators, desiccants, mycotoxins (Fusarium toxins, Ochratoxin A and ergot alkaloids), pesticides, heavy metals, dioxins and chlorates.

The overwhelming majority of samples complied with legal and guideline limits. Levels of applied storage pesticides, growth regulators and desiccants were all within legal limits and levels did not vary significantly from season to season. There does, however, appear to be an increase in the use of the synthetic pyrethroids cypermethrin and deltamethrin and a reduction in the application of the organophosphate pirimiphos methyl.

The storage mycotoxin, Ochratoxin A was generally detected only at low concentrations, with mean and median values largely being determined at levels at, or just above the LOQ (0.1 µg/kg). This demonstrates that mould growth and toxin synthesis are being adequately controlled by suitable storage conditions. Levels of Fusarium mycotoxins, which in previous surveillance projects have showed significant seasonal variations, increased from 2011 to 2012 but then largely declined from the 2012 to 2015. Ergot alkaloids were generally determined at very low levels, the vast majority of samples having a median value below the method LOQ.

Selected elements were surveyed during three harvest periods on selected sample types.
Dioxins were analysed on selected AIC samples from the 2014 and 2015 harvests.

2. Introduction

The aim of this project was to investigate the occurrence of key contaminants in UK-grown wheat, barley and oats and their co-products to demonstrate safety for use for milling, malting and animal feed, as well as the extent of compliance with legal and guideline limits. Throughout the project, a “horizon scanning” exercise was carried out, looking at legislation, publications from official bodies such as the UK Food Standards Agency, the European Food Safety Authority (EFSA), and the World Health Organisation’s Joint Expert Committee on Food Additives (JECFA), as well as the scientific, agricultural and medical press, in order to identify emerging issues and trends. The contaminants investigated were selected based on this literature survey, and consultation with a steering committee consisting of representatives of the relevant Trade Associations (the Agricultural Industries Confederation (AIC), the National Association of British and Irish Millers (nabim) and the Maltsters’ Association of Great Britain (MAGB), the AHDB and scientists from Campden BRI. Samples of milling and feed wheat, malting and feed barley, feed oats, wheatfeed and oatfeed were collected from harvests 2012, 2013, 2014 and 2015, either immediately after harvest, or after a period of storage. Contaminants sought included:

- Mycotoxins – Ergot alkaloids, Ochratoxin A, Fusarium toxins
- Pesticides – storage insecticides, growth regulators and desiccants
- Metals – lead, cadmium, arsenic, mercury, nickel and copper
- Dioxins
- Chlorates

The principal contaminants affecting the safety and acceptability of UK grown cereals are mycotoxins. Several HGCA/AHDB projects over the past fifteen years (Baxter, 2003, 2006a, 2006b; Salmon, 2006, Baxter *et al.*, 2009; Slaiding *et al.*, 2013; HGCA, 2011, AHDB projects; PR500 2012, SR23 2013, PR547 2015, PR549 2015 and PR555 2016), have accumulated data on a range of mycotoxins.

A key group of mycotoxins are those produced by the various Fusarium species associated with Fusarium head blight. Fusarium species infect barley, wheat and oats during growth, particularly at the flowering stage, and produce a range of mycotoxins. The principal mycotoxins found are the trichothecenes, of which deoxynivalenol (DON) is the most common and the most widely studied. Data from the above projects indicate that levels of DON in UK cereals are generally well below legal limits and also lower than in other countries. However, incidence of DON varies considerably depending on climatic conditions and agronomic practice and crop losses due to Fusarium contamination remain significant. Continued monitoring remains essential to assure crop quality and also in understanding the incidence of contamination and the efficacy of control measures. Although DON is the predominant trichothecene in grain, some of the other trichothecenes have greater toxicity, and hence, it is important that they are also monitored. T-2 and HT-2 are Type A trichothecenes, which are thought to be produced predominantly by *F. sporotrichioides* and *F. langsethiae*. T-2 and HT-2 have been widespread in raw oats for many years and more recently, have been found in barley and wheat. The European Food Safety Authority (EFSA) has published a scientific opinion (EFSA, 2011b) on the risks for animal and public health related to the presence of T-2 and HT-2 toxins in food and feed. This established a TDI of 100 ng/kg b.w. for the sum of T-2 and HT-2 and concluded that at current human dietary intakes, they do not present a concern to health. No legal limits have yet been set but the European Commission (EC, 2013) has set indicative levels for T-2 and HT-2 in cereals and cereal products with a recommendation that repetitive findings above these levels should be investigated by Member States and feed and food business operators with the objective to obtain more information on the different factors, including agronomic factors, which lead to relative high levels of T-2 and HT-2 toxin in cereals and cereal products. Data from previous AHDB projects indicate that incidence of these toxins in the UK increased significantly between 2005 and 2010. Continued monitoring was included in this project to establish whether this trend persisted. Incidence appears to be highly weather-related and more data is required to further understand the occurrence of these toxins.

Nivalenol (NIV) is produced by the same Fusarium species as DON, though probably by different isolates, and also by *F. Poae*. EFSA have published an opinion on the presence of nivalenol in food and feed (EFSA, 2013a). The highest mean concentrations for nivalenol in food, feed and unprocessed grains were observed in oats, maize, barley and wheat and products thereof. Higher concentrations were observed in unprocessed grains compared to grains for human consumption. The CONTAM Panel established a tolerable daily intake (TDI) of 1.2 µg/kg b.w. per day; chronic dietary exposures, based on the available occurrence data in food, were below the TDI and were, therefore, not of health concern. Nevertheless, nivalenol was included in the monitoring. Other trichothecenes found in cereals include neosolaniol (commonly found on oats) and diacetoxyscirpenol (on which the EC asked EFSA for a scientific opinion in 2012). Thus, there is likely to be a continuing need for data on the incidence of these toxins.

Zearalenone (ZON) is another mycotoxin produced predominantly by *F. culmorum* and *F. graminearum*. It differs from the trichothecenes in being predominantly produced late in the crop growing season, close to harvest. Though not widespread in UK cereals, high levels were found in 2008 and to a lesser extent on 2009 when wet weather delayed harvest in some areas. ZON can affect the health and productivity of livestock. Legal limits for animal feed are relatively low and could be exceeded in some years. In an EFSA Opinion on the presence in food (EFSA, 2011d), estimates of chronic dietary exposure to zearalenone, based on the available occurrence data, were at or below the TDI for all age groups and were considered not to be a health concern. EFSA have been asked for an opinion on the risks to animal health related to its presence in animal feed (EFSA, 2015d).

Ergot alkaloids

Ergot (*Claviceps purpurea*) is an important disease of cereals which can lead to extensive financial losses to growers due to the toxicity of ergot present in the grain. Ergot levels vary from year to year, and are influenced by weather at flowering, which affects both the host and the pathogen. Ergot is also the name given to the black fungal bodies or sclerotia that replace the grain in the ear. Ergot kernels are hard, purplish- black dense tuber-like bodies, 2 - 22mm long, which can be easily seen on visual inspection of the grain. Until recently, there were no national legal limits for ergot in EU member states but there were commercial limitations in some countries. For example, in the UK, standards for ergot are 0.001% ergot by weight for feed grain and a zero tolerance for all other grain (HGCA, 2002).

Controls based on sclerotia have significant limitations; determination of the contamination rate is often inaccurate, the composition and toxicity of the sclerotia are variable and it is impossible to detect (and therefore, to remove) sclerotia in processed feedstuffs. It has, therefore, been suggested that the current limits on sclerotia should be replaced by chemical analysis of the ergolines (EFSA, 2005). In early 2012, the EC recommended monitoring of ergot alkaloids in feed and food with a focus on the six predominantly present ergot alkaloids, i.e. ergometrine, ergotamine, ergosine, ergocristine, ergocryptine and ergocornine and their related –inines (EC, 2012). A survey published by EFSA (EFSA, 2011c) found several different alkaloids at trace levels in a variety of rye and wheat based food and feed samples.

EFSA issued a Scientific Opinion on ergot alkaloids in 2012 (EFSA, 2012). The Panel noted that the available data suggested that there was not a marked difference between the potency of the different ergot alkaloids and, therefore, they derived group values for toxicity indices. EFSA then derived a group TDI (Tolerable Daily Intake) of 0.6 µg/kg body weight/ day and a group ARfD (acute reference dose) of 1 µg/kg body weight.

Data on 1,716 samples of food, 496 samples of animal feed and 67 samples of unprocessed grains of unknown end-use were reviewed by EFSA (EFSA, 2012). Ergotamine, ergocristine, ergosine and ergocornine were generally more abundant than α- and β-ergocryptine and ergometrine. Most of the food samples were non- or minimally processed foods, mainly grain milling products and the majority of these were rye and wheat-based. For the whole grain and grain based foods, less than 20% contained quantifiable amounts of ergot alkaloids. Amongst the grain milling products, almost 50% contained quantifiable amounts of ergot alkaloids, but the actual concentrations were much lower than those in whole grain. Grain sorting and cleaning methods in general tend significantly to reduce contamination with ergot alkaloids further down the food chain. Milling processes, on the other hand, tend to result in redistribution of sclerotia and alkaloids between the different fractions.

Data from AHDB projects are largely consistent with the EFSA data. Further data is required on levels of ergot alkaloids in grain samples and processed products where contamination with sclerotia is not readily evident. The EC (EC, 2015) has set a limit on ergot sclerotia in unprocessed cereals, except for corn and rice, and intends to set limits for the sum of the 12 ergot alkaloids in cereals and cereal products by 1 July 2017.

The mycotoxins described above are all predominantly formed during growth in the field. A second very important issue is those mycotoxins formed during grain storage. Ochratoxin A (OTA) is the principal mycotoxin found in stored cereals such as wheat and barley and is mainly produced by *Penicillium* and *Aspergillus* mould species. Both of these moulds are widespread contaminants of cereals in temperate climates, particularly in Europe, but rarely colonise the growing crop. They invade grain mainly during storage and can grow rapidly given suitable conditions of temperature and moisture. Infection with *Penicillium verrucosum* during storage is the most common source of OTA in barley, wheat and oats. Analytical data over the past fifteen years show that almost all samples taken from stores comply with either legal limits for food or the guideline levels for feedstuffs. However, mould infections are frequently discontinuous in stored grain, often due to localised damp areas within a bulk (“hot spots”) and occasional high levels of ochratoxin A are found. Continued monitoring of ochratoxin A is necessary to ensure control measures remain effective.

Pesticide residues

A broad range of herbicides, fungicides and insecticides are used in cereal production. The EU and many other non-EU countries operate strict maximum limits for pesticide residues in cereals and other food crops and evidence of compliance with these limits is an important factor for both domestic and export markets. In preceding monitoring projects, analysis has focussed on insecticides used in grain stores, either as treatments for the fabric of the store or sprayed on to the grain itself. A number of organophosphate insecticides have been used over the years; these have largely been phased out but a few remain in use, in particular, in countries exporting grain to the UK. These have now largely been replaced by synthetic pyrethroids, in particular, deltamethrin. Stored whole grains were tested for a range of insecticides currently or recently approved for use on stored cereal grain or in cereal stores in the UK.

The herbicide glyphosate is used in cereal production as a desiccant close to harvest time where it promotes grain ripening and assists drying down of the crop and efficiency of harvesting. It is particularly used when the harvest period is wet. Approval for use of glyphosate in Europe expired at the end of 2015 but was extended to June 2016 while re-assessment took place. The European Commission then proposed the re-approval of the glyphosate for a further 15 years. EFSA's current opinion on glyphosate, issued in November 2015, is that it is unlikely to cause cancer. However, the International Agency on Research on Cancer (IARC) classifies it as probably carcinogenic to humans and a resolution passed by the European Parliament subsequently proposed that it should only be approved, with new restrictions, for another 7 years. In the event, in June 2016, approval was extended only until December 2017. Monitoring was carried out in the last three years of the project.

The growth regulator chlormequat is widely used on cereals, either alone or in combination with mepiquat, to restrict stem elongation and reduce the risk of lodging (which can cut yield and increase the likelihood of mould growth and mycotoxins contamination). It has been cited as one of the most common residues detected on cereals by several EU member states including the UK (EC 2005a). Both chlormequat and mepiquat are systemic and water-soluble, and can, therefore, readily persist into products such as beer. This means that monitoring residue levels is particularly important in malting barley and malt.

Elements

Limits are set in the EU for lead, cadmium, arsenic and mercury in cereals for food and feed use. Previous studies have shown UK cereals to be compliant with these limits but there is little recent published data and consequently, samples from the 2011 harvest were analysed for cadmium, lead, aluminium, arsenic and mercury.

In 2009, EFSA set a reduced tolerable weekly intake (TWI) for cadmium of 2.5µg/kg body weight and the European Commission has subsequently proposed reductions in the maximum levels allowed in certain foodstuffs. In 2014 the European Commission published Recommendation 2014/193/EU on the reduction of the presence of cadmium in foodstuffs. All foods contain low levels of cadmium, with the highest concentrations being found in shellfish and offal. Some types of cereals, particularly hard wheats used for breadmaking, are considered to be more at risk of taking up cadmium from the soil, thus bread is often a major source of cadmium in the diet. In previous AHDB monitoring projects, cadmium levels have largely been well within legal limits for food but the European Commission has recently reviewed the maximum permitted levels of cadmium in certain foodstuffs, including barley and wheat, with a view to possible reductions. This might lead to a higher proportion of the wheat crop coming close to or breaching the limit. Monitoring is necessary to establish if this is the case.

Lead is not readily taken up by cereals; consequently, levels in grain are not related to the levels in soils except in heavily contaminated areas. The most likely sources of contamination are from atmospheric deposition due to traffic emissions, which have fallen substantially in Europe since the introduction of lead-free fuel, and from industrial pollution. It is appropriate to establish if this decline in usage is reflected in levels in grain. Limits for lead in cereals are set for grain for food use (EC, 2006c) and for use in animal feed (EC, 2002). Limits are also set for arsenic and mercury and, under pesticides legislation, for copper (EC, 2005b).

The European Commission has recently recommended that Member States monitor the presence of nickel in foods - including cereal grains and cereal based products - during 2016 – 2018 (EC, 2016). Cereals and cereal-based products were amongst the foodstuffs identified as requiring monitoring.

Dioxins

Potentially, cereals can become contaminated with dioxins as a result of aerial deposition and background contamination of soils. Levels may be higher close to point sources of emission such as incinerators or industrial processing plants. However, since dioxins are fat-soluble, plant products with a low fat content contain significantly lower levels of dioxins than do foods of animal origin. In many cases, concentrations of most of the congeners are very low, often below the limit of detection. This means that reported levels vary significantly depending upon the limit of determination of the method used, and whether the results are reported as lower- medium- or upper bound levels. Dioxin analysis is extremely expensive and most surveys concentrate on animal products or fats and oils, which are the most likely sources of contamination. Consequently, there is little data available for cereals. Out of several hundred analyses reported in the EU's SCOOP report on dioxins in the diet, only 11 related to cereals or cereal products (EC, 2000). Overall, the levels of dioxins in cereals and other vegetables were found to be very low, often close to the limit of determination.

In the UK, three composite samples of malting barley from the 1999 harvest gave upper bound dioxin concentrations ranging from 0.11 – 0.16 pg WHO TEQs /g. These values were in the same range as those quoted in the SCOOP report. In 2015, EFSA were asked for an opinion on dioxins and dioxin-like PCBs; publication is expected in mid-2017.

A recent European Food Safety Authority Opinion has specifically identified a need for more data on chlorate levels for some foods. The current default residue limit for chlorate of 0.01 mg/kg in cereals was set when chlorate was used as a pesticide, but it does not take into account other legal uses, for example, as a disinfectant. The World Health Organisation has set a guideline level of 700 µg/L in drinking water. Resolving the disparity between the two limits requires more data on levels in foodstuffs and since water is used in processing of some cereal products and notably in the malting process, a small amount of monitoring of malting barley and malt was included in the project.

3. Materials and methods

3.1. Samples

A sampling and analytical programme was developed by the steering committee comprising AHDB, the trade associations (nabim, MAGB and AIC) and representatives from Campden BRI.

This committee met prior to each harvest period. Additional meetings took place between these periods to discuss emerging issues and sampling schedules.

Samples of (1) commercial milling wheat, feed wheat, wheatfeed; (2) malting barley and malts prepared from those barleys, and (3) feed barley, feed oats and oatfeed were taken by members of the trade associations and sent to Campden BRI for analysis.

Sampling plans were designed by each of the three trade associations and were intended to give a broad geographical spread representative of the UK market.

Two main tranches were collected each year:

- At delivery immediately after harvest (September): these were analysed for Fusarium toxins and ergot alkaloids. Selected samples were also analysed for elements, dioxins, plant growth regulators and glyphosate
- The second main tranche were collected from grain stores after 6 months storage (March): these were analysed for Ochratoxin A and storage pesticides

An additional tranche of stored milling wheat samples were collected in January for OchratoxIn A analysis. Paired samples of malting barley and malt were collected between November and March. These samples were analysed for Fusarium toxins and Ochratoxin A. The barley samples were additionally analysed for pesticide residues.

Table 1: Samples collected - 2012 harvest

Cereal	Date collected	Samples	Analytes
Malting barley	Sep-12		Trichothecenes, Zearalenone, Ergot Alkaloids, Heavy Metals
Feed barley	Sep-12		Trichothecenes, Zearalenone, Ergot Alkaloids, Heavy Metals
Milling wheat	Sep-12		Trichothecenes, Zearalenone , Ergot Alkaloids, Plant Growth Regulators
Feed wheat	Sep-12		Trichothecenes, Zearalenone , Ergot Alkaloids
Wheatfeed	Sep-12		Trichothecenes, Zearalenone, Ergot Alkaloids
Feed oats	Sep-12		Trichothecenes, Zearalenone , Ergot Alkaloids, Heavy Metals
Oatfeed	Sep-12		Trichothecenes, Zearalenone, Ergot Alkaloids, Heavy Metals
Malting barley	Nov 12 - Mar 13		Trichothecenes, Zearalenone, Ergot Alkaloids, Ochratoxin A, Glyphosate, Plant Growth Regulators, Pesticides
Malted barley	Nov 12- Mar 13		Trichothecenes, Zearalenone & Ochratoxin A
Feed barley	Mar-13		Ochratoxin A
Milling wheat	Jan-13		Ochratoxin A, Pesticides
Milling wheat	Mar-13		Ochratoxin A, Pesticides
Feed wheat	Mar-13		Ochratoxin A, Pesticides
Wheatfeed	Mar-13		Ochratoxin A, Pesticides
Feed oats	Mar-13		Ochratoxin A, Pesticides
Oatfeed	Mar-13		Ochratoxin A

Table 2: Samples collected – 2013 harvest

Cereal	Date collected	Samples	Analytes
Malting barley	Sep-13		Trichothecenes, Zearalenone, Ergot Alkaloids
Feed barley	Sep-13		Trichothecenes, Zearalenone, Ergot Alkaloids
Milling wheat	Sep-13		Trichothecenes, Zearalenone , Ergot Alkaloids, Glyphosate, Plant Growth Regulators
Feed wheat	Sep-13		Trichothecenes, Zearalenone , Ergot Alkaloids
Wheatfeed	Sep-13		Trichothecenes, Zearalenone, Ergot Alkaloids
Feed oats	Sep-13		Trichothecenes, Zearalenone , Ergot Alkaloids
Oatfeed	Sep-13		Trichothecenes, Zearalenone, Ergot Alkaloids
Malting barley	Nov 13 - Mar 14		Trichothecenes, Zearalenone, Ergot Alkaloids, Ochratoxin A, Pesticides
Malted barley	Nov 13 - Mar 14		Trichothecenes, Zearalenone, Ochratoxin A
Feed barley	Mar-14		Ochratoxin A, Pesticides
Milling wheat	Jan-14		Ochratoxin A, Pesticides
Milling wheat	Mar-14		Ochratoxin A, Pesticides
Feed wheat	Mar-14		Ochratoxin A, Pesticides
Wheatfeed	Mar-14		Ochratoxin A, Pesticides
Feed oats	Mar-14		Ochratoxin A, Pesticides
Oatfeed	Mar-14		Ochratoxin A, Pesticides

Table 3: Samples collected – 2014 harvest

Cereal	Date collected	Samples	Analytes
Malting barley	Sep-14		Trichothecenes, Zearalenone, Ergot Alkaloids, Heavy Metals
Feed barley	Sep-14		Trichothecenes, Zearalenone, Ergot Alkaloids, Dioxins
Milling wheat	Sep-14		Trichothecenes, Zearalenone , Ergot Alkaloids, Glyphosate, Plant Growth Regulators, Heavy Metals
Feed wheat	Sep-14		Trichothecenes, Zearalenone , Ergot Alkaloids, Heavy Metals, Dioxins
Wheatfeed	Sep-14		Trichothecenes, Zearalenone, Ergot Alkaloids, Heavy Metals
Feed oats	Sep-14		Trichothecenes, Zearalenone , Ergot Alkaloids
Oatfeed	Sep-14		Trichothecenes, Zearalenone, Ergot Alkaloids
Malting barley	Nov 14 - Mar 15		Trichothecenes, Zearalenone, Ergot Alkaloids, Ochratoxin A, Pesticides
Malted barley	Nov 14 - Mar 15		Trichothecenes, Zearalenone, Ochratoxin A
Feed barley	Mar-15		Ochratoxin A, Pesticides
Milling wheat	Jan-15		Ochratoxin A, Pesticides
Milling wheat	Mar-15		Ochratoxin A, Pesticides
Feed wheat	Mar-15		Ochratoxin A, Pesticides
Wheatfeed	Mar-15		Ochratoxin A, Pesticides
Feed oats	Mar-15		Ochratoxin A, Pesticides
Oatfeed	Mar-15		Ochratoxin A, Pesticides

Table 4: Samples collected – 2015 harvest

Cereal	Date collected	Samples	Analytes
Malting barley	Sep-15		Trichothecenes, Zearalenone, Ergot Alkaloids, Heavy Metals
Feed barley	Sep-15		Trichothecenes, Zearalenone, Ergot Alkaloids
Milling wheat	Sep-15		Trichothecenes, Zearalenone , Ergot Alkaloids Glyphosate, Plant Growth Regulators, Heavy Metals
Feed wheat	Sep-15		Trichothecenes, Zearalenone , Ergot Alkaloids, Dioxins
Wheatfeed	Sep-15		Trichothecenes, Zearalenone, Ergot Alkaloids
Feed oats	Sep-15		Trichothecenes, Zearalenone , Ergot Alkaloids
Oatfeed	Sep-15		Trichothecenes, Zearalenone, Ergot Alkaloids
Malting barley	Nov 15 - Mar 16		Trichothecenes, Zearalenone, Ergot Alkaloids, Ochratoxin A, Pesticides, Chlorates
Malted barley	Nov 15 - Mar 16		Trichothecenes, Zearalenone, Ochratoxin A , Chlorates
Feed barley	Mar-16		Ochratoxin A, Pesticides
Milling wheat	Jan-16		Ochratoxin A, Pesticides
Milling wheat	Mar-16		Ochratoxin A, Pesticides
Feed wheat	Mar-16		Ochratoxin A, Pesticides
Wheatfeed	Mar-16		Ochratoxin A, Pesticides
Feed oats	Mar-16		Ochratoxin A, Pesticides
Oatfeed	Mar-16		Ochratoxin A, Pesticides

3.2. “Horizon scanning” for emerging issues

Databases comprising information from scientific bodies, national surveillance programmes, global rapid alerts, and industry trade bodies were regularly updated in order to identify potential emerging issues. This information was used by the steering committee throughout the project in order to make cost effective judgements on which analytes to include within the surveillance programme.

3.3. Analysis of mycotoxins

All ergot alkaloids were analysed at the Chipping Campden site. Barley (malting and feed) and oats were analysed at the Nutfield site, while those in wheat (milling and feed) were analysed at the Chipping Campden site.

Ergot Alkaloids

Six ergot alkaloids, namely ergotamine, ergometrine, ergosine, ergocryptine, ergocristine, ergocornine, and the corresponding –inine epimers were analysed by a LC-MS/MS procedure based on a published method (R Krska and C Crews, 2007).

Ground samples were extracted into acetonitrile/ammonium carbonate buffer and cleaned-up by dispersive SPE prior to LC-MS/MS determination. The limit of quantification for each ergot alkaloid and epimer was 1 µg/kg. The method is accredited to ISO17025:2005.

Fusarium toxins: trichothecenes and zearalenone

Trichothecenes (deoxynivalenol, 3- and 15- acetyl-DON, nivalenol, neosolaniol, diacetoxyscirpenol, fusarenone-X, T-2 toxin and HT-2 toxin) were analysed by three in-house procedures.

Wheat samples were analysed by a LC-MS/MS procedure. Samples were ground to a fine powder and trichothecenes extracted using acetonitrile/water, then partially purified using trichothecene clean-up columns, then separated and quantified by liquid chromatography-mass spectrometry. The limit of quantification for each trichothecene in this procedure was 10 µg/kg. The method is accredited to ISO17025:2005.

Barley and oat samples were analysed by a GC-MS procedure based on a published method (Patel *et al..,* 1996). Samples were ground to a fine powder, trichothecenes were extracted using acetonitrile/water, then partially purified using trichothecene clean-up columns, derivatised and analysed by GC-MS. The limit of quantification for each trichothecene was 5 µg/kg.

For paired barley and malted barley samples, a separate, a more sensitive method was used for T-2 and HT-2 toxins. Samples were ground to a fine powder, trichothecenes extracted using methanol/water, and then purified using specific immunoaffinity columns, separated and quantified by liquid chromatography-mass spectrometry. The limit of quantification for each trichothecene in this procedure was 1 µg/kg. The method is accredited to ISO17025:2005.

Zearalenone was analysed by in-house procedures.

Wheat samples were analysed by a LC-MS/MS procedure. Samples were ground to a fine powder, then after extraction with acetonitrile/water, the extracts were filtered then the zearalenone separated and quantified by liquid chromatography-mass spectrometry.

Barley and oat samples were analysed by a HPLC-FD method. Samples were ground to a fine powder then after extraction with acetonitrile/water, specific immunoaffinity columns were used for the clean-up stage. Detection and quantification were achieved by HPLC with fluorescence detection.

The limit of quantification for both methods was 2 µg/kg. Both sites are accredited to ISO17025:2005 for this analysis.

Ochratoxin A

Ochratoxin A was analysed by two in-house procedures.

Samples were solvent extracted and cleaned up by immunoaffinity columns. Detection and quantification were carried out by HPLC with fluorescence detection. The limit of quantification was 0.1 µg/kg. Both sites are accredited to ISO17025:2005 for this analysis.

3.4. Analysis of pesticide residues

Storage pesticides

Pesticides were determined by an in-house method. After extraction into acetonitrile, the extract was purified by dispersive SPE prior to determination by GC-MS/MS and LC-MS/MS. The limit of quantification was 0.01 mg/kg for each residue tested.

Table 5: Pesticide residue screen

Organochlorine Analytes

α HCH
β HCH
γ HCH (lindane)
2,4' – DDE
2,4' – DDT
2,4' – TDE
4,4' – DDE
4,4' – DDT
4,4' – TDE
Aldrin
Captan
Dicofol
Dieldrin
Endosulfan
Endrin
Heptachlor
Heptachlor epoxide
Hexachlorobenzene
Quintozene
Tecnazene
Tetradifon
Vinclozolin

Organophosphates

Azinphos methyl
Bromophos
Bromophos-ethyl
Cadusafos
Chlorfenvinphos
Chlorpyrifos
Chlorpyrifos-methyl
Diazinon
Dichlorvos
Dimethoate
Ethion
Ethoprophos
Etrimfos
Fenamiphos
Fenitrothion
Fonofos
Heptenophos
Malaoxon
Malathion
Mecarbam
Methacrifos
Methidathion
Mevinphos
Parathion
Parathion-methyl
Phenthoate
Phosalone
Phosmet
Phosphamidon
Pirimiphos-ethyl
Pirimiphos-methyl
Profenofos
Prothiophos
Propetamphos
Pyrazophos
Pyridaphenthion
Quinalphos
Tetrachlorvinphos
Tolclofos-methyl
Triazophos

Carbamates

- Bendiocarb
- Carbaryl
- Chlorpropham
- Pirimicarb
- Propoxur

Synthetic Pyrethroids

- Bifenthrin
- Cyfluthrin
- Cyhalothrin (λ)
- Cypermethrin
- Cyprodinil
- Deltamethrin
- Fenpropathrin
- Fenvalerate
- Permethrin

Miscellaneous Analytes

- 2-phenylphenol
- 3-hydroxycarbofuran
- Acetamiprid
- Aldicarb
- Aldicarb sulphone
- Aldicarb sulphoxide
- Aminocarb
- Anthraquinone
- Atrazine
- Azoxystrobin
- Benalaxyl
- Biphenyl
- Bitertanol
- Boscalid
- Bromacil
- Bromopropylate
- Bromuconazole
- Bupirimate
- Buprofezin
- Carbendazim
- Carbetamide
- Carbofuran
- Carboxin
- Chloramben
- Chloridazon
- Chloroxuron
- Cyanazine
- Dazomet
- Dichlofluanid
- Dicloran
- Difenoconazole
- Diflubenzuron
- Dimefuron
- Dimethomorph
- Diniconazol
- Diphenylamine
- Diuron
- Ethidimuron
- Ethiofencarb
- Ethofumesate
- Furalaxyl
- Hexythiazox
- Imazalil
- Imidacloprid
- Iprobenphos
- Iprodione
- Isofenphos methyl
- Isoproturon
- Kresoxim methyl
- Linuron
- Metalaxyl
- Metazachlor
- Methabenzthiazuron
- Methamidophos
- Methiocarb
- Methomyl
- Metobromuron
- Metolachlor
- Monolinuron
- Myclobutanil
- Oxadixyl
- Oxamyl
- Oxycarboxin
- Penconazole
- Pendimethalin
- Piperonyl butoxide
- Prochloraz
- Procymidone
- Prometryn
- Propamocarb hydrochloride
- Propargite
- Propazine
- Propiconazole
- Propyzamide
- Pyriproxyfen
- Simazine
- Spinosad
- Tebuconazole
- Terbuthylazin-desethyl
- Thiabendazole

Fenarimol
Fenhexamide
Fenpyroximate
Fludioxonil
Flusilazole
Flutolanil
Thiamethoxam
Thiophanate methyl
Triadimenol
Trifloxystrobin
Trifluralin

Glyphosate

Glyphosate was analysed by solvent extraction with stable isotope dilution. Detection and quantification was by LC-MS/MS *(Granby et al., 2003).* The limit of quantification was 0.05 mg/kg.

Growth regulators

Chlormequat and mepiquat were analysed by solvent extraction followed by separation, detection and quantification by LC-MS/MS. The method was based on a published procedure *(Vahl et al., 1998)*. The limit of quantification was 0.01 mg/kg.

3.5. Analysis of elements

All metal assays were carried out at the Chipping Campden site. Samples of grain were digested in a microwave digestion oven at elevated pressure in the presence of nitric acid. The diluted digest was introduced into an inductively-coupled plasma mass spectrometer (ICP-MS). The individual isotopes were determined by their response relative to that obtained from standard solutions. The method is accredited to ISO17025:2005.

3.6. Analysis of chlorates

Chlorate was analysed by solvent extraction, stable isotope dilution, detection and quantification by LC-MS/MS.

3.7. Analysis of dioxins

Dioxins assays were sub-contracted to an ISO17025:2005 accredited laboratory. Determination was by high resolution GC/MS.

4. Results

4.1. Field mycotoxins

Deoxynivalenol

Results for milling wheat and malting barley are shown in Tables 6 and 7. The maximum and median levels of deoxynivalenol determined in milling wheat in this four year study were higher than those determined in the previous project (RD-2008-3572) but comparable to levels from the 2006-08 harvests.

Incidence and mean levels in wheat declined over the four harvest periods. The highest levels were determined in the 2012 harvest. Five samples exceeded the maximum level (1250 µg/kg). No further exceedances were determined in the following three years of harvest samples.

Incidence, mean and maximum levels in malting barley were lower than milling wheat for the corresponding harvest seasons, with mean and maximum levels declining from 2012 to 2015. This reduced incidence, and lower residue levels of deoxynivalenol in barley have been regularly observed in previous projects.

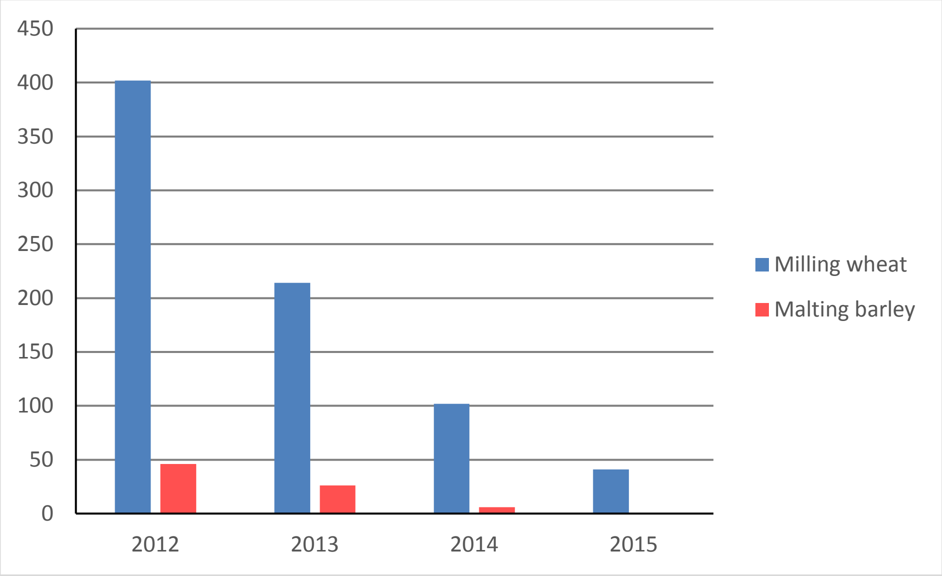
Table 6: Deoxynivalenol in freshly harvested milling wheat

Harvest year	Incidence	Mean	Maximum	Minimum	Median	Mode
	%	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
2012	100	402	2780	24	210	200
2013	99	214	1040	<10	122	37
2014	92	102	755	<10	58	<10
2015	51	41	632	<10	10	<10

Table 7: Deoxynivalenol in freshly harvested malting barley

Harvest year	Incidence	Mean	Maximum	Minimum	Median	Mode
	%	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
2012	50	46	214	<5	<5	<5
2013	51	26	204	<5	9	<5
2014	38	6	33	<5	<5	<5
2015	20	<5	19	<5	<5	<5

Figure 1: Mean value (µg/kg) of DON in food grains 2012-15



Results for feed grains are shown in Table 8. Similar to food grains, the incidence, mean and maximum levels for feed wheat, wheatfeed and oatfeed were higher than those determined for the 2009-11 harvests in the previous survey, RD-2008-3572. There is however, little correlation between the levels of DON in feed and food grains from the same harvests. The highest levels in feed wheat, wheatfeed and oatfeed were all determined in the 2012 harvest. Feed barley and feed oat levels remained largely the same throughout the project. All samples were within EC guideline levels for feedingstuffs. As with the food grains incidence, mean and maximum levels were higher in wheat than barley.

Table 8: Deoxynivalenol in cereal feedingstuffs

Sample	Harvest Year	Incidence	Mean	Maximum	Minimum	Median	Mode
		%	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
Feed Wheat	2012	100	391	883	101	212	N/A
	2013	100	159	400	20	160	N/A
	2014	60	61	243	<10	55	<10
	2015	70	29	144	<10	11	<10
Wheatfeed	2012	100	846	1668	316	776	N/A
	2013	100	595	1000	260	495	N/A
	2014	100	242	650	48	225	N/A
	2015	100	187	607	12	174	N/A
Feed barley	2012	11	10	42	<5	<5	<5
	2013	78	95	377	<5	36	<5
	2014	55	13	44	<5	5	<5
	2015	47	14	91	<5	<5	<5
Feed oats	2012	8	38	400	<5	<5	<5
	2013	77	81	334	<5	62	54
	2014	58	36	309	<5	6	N/A
	2015	27	15	67	<5	<5	<5
Oatfeed	2012	50	224	1313	<5	7	<5
	2013	100	120	332	15	54	<5
	2014	90	155	859	<5	81	<5
	2015	92	55	92	<5	50	N/A

Figure 2: Mean value (µg/kg) of DON in cereal feedingstuffs 2012-15

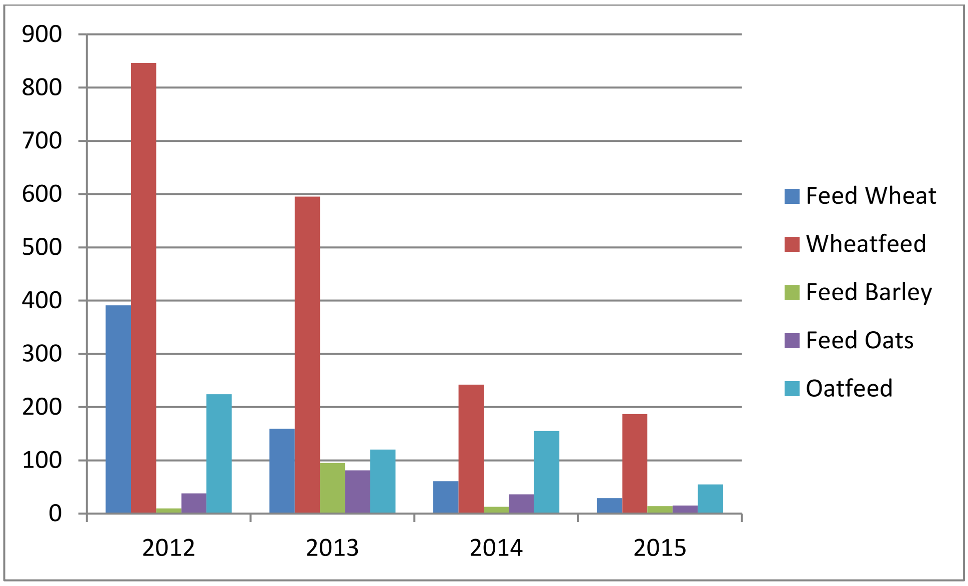


Table 9: Deoxynivalenol in malting barley and malt

Harvest year	Sample	Incidence	Mean	Maximum	Minimum	Median	Mode
		%	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
2012	Barley	62	78	555	<5	32	<5
2012	Malt	43	77	746	<5	<5	<5
2013	Barley	59	14	48	<5	<5	<5
2013	Malt	50	15	97	<5	5	<5
2014	Barley	19	<5	42	<5	<5	<5
2014	Malt	25	5	29	<5	<5	<5
2015	Barley	10	<5	11	<5	<5	<5
2015	Malt	0	<5	<5	<5	<5	<5

A second set of malting barleys was collected each year, together with a sample of the malt produced from each barley sample. Results for these paired samples are shown in Table 9. There was little correlation between incidence and maximum levels in individual barley and malt pairs over the four harvest periods, although mean levels were comparable.

T-2 and HT-2 toxins

T-2 and HT-2 toxins are mycotoxins produced by various Fusarium species. T-2 toxin is rapidly metabolised to a large number of products, HT-2 toxin being a major metabolite. They are generally treated as a pair when considering incidence and regulatory levels hence all results are presented as the sum of the two toxins.

Table 10: Indicative levels for the sum of T-2 and HT-2 in cereals and cereal products

Matrix	T-2 +HT-2 (µg/kg)
Unprocessed malting barley	200
Unprocessed wheat	100
Cereal products for feed and compound feed	
Oat milling products (husks)	2000
Other cereal products	500
Compound feed	200

Results for the freshly harvested milling wheat and malting barley are shown in Tables 11 and 12. T-2 and HT-2 were rarely detected in wheat samples. This is in line with historic patterns from previous projects. Malting barley was more prone to contamination, but even here, incidence, mean and median levels were generally very low. The highest level determined to date, 486 µg/kg, was in a malting barley sample from the 2012 harvest. Incidence from this harvest was very low (3%), with median and mode values both below the method LOQ (5 µg/kg).

Table 11: T-2 + HT-2 in freshly harvested milling wheat

Harvest year	Incidence	Mean	Maximum	Minimum	Median	Mode
	%	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
2012	2	<10	27	<10	<10	<10
2013	3	<10	50	<10	<10	<10
2014	0	<10	<10	<10	<10	<10
2015	0	<10	<10	<10	<10	<10

Table 12: T-2 + HT-2 in freshly harvested malting barley

Harvest year	Incidence	Mean	Maximum	Minimum	Median	Mode
	%	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
2012	3	21	486	<5	<5	<5
2013	41	13	59	<5	<5	<5
2014	14	<5	24	<5	<5	<5
2015	18	6	51	<5	<5	<5

The paired barley and malt samples were analysed by a LC-MS/MS method with greater sensitivity (a quantification limit of 1 µg/kg rather than 10 µg/kg). This led to a generally greater level of incidence of contamination (Table 13) but also to lower mean values in the barleys. Other than the 2012 harvest data, there was clear evidence that levels and incidence were generally lower in malt than in the corresponding barley.

Table 13: T-2 + HT-2 in malting barley and malt pairs

Harvest year	Sample	Incidence	Mean	Maximum	Minimum	Median	Mode
		%	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
2012	Barley	16	2	6	<2	<2	<2
2012	Malt	0	<2	<2	<2	<2	<2
2013	Barley	50	9	51	<2	3	<2
2013	Malt	18	2	21	<2	<2	<2
2014	Barley	56	5	15	<2	4	<2
2014	Malt	19	<2	3	<2	<2	<2
2015	Barley	80	5	10	<2	4	<2
2015	Malt	20	<2	5	<2	<2	<2

The incidences of T-2 and HT-2 in feed wheat and feed barley (Table 14) matched those of the food grain. There was widespread contamination of feed oats and oatfeed which was comparable to previous surveys. As anticipated, due to the higher husk content, and in line with previous years' data, the highest levels were determined in oatfeed samples. Mean and maximum levels for the oatfeed samples over the four year surveillance programme were higher than those determined in the previous three years. The highest level, 4310 µg/kg, was determined in 2014 harvest samples.

Table 14: T-2 + HT-2 in cereal feedingstuffs

Sample	Harvest Year	Incidence	Mean	Maximum	Minimum	Median	Mode
		%	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
Feed Wheat	2012	0	<10	<10	<10	<10	<10
	2013	0	<10	<10	<10	<10	<10
	2014	0	<10	<10	<10	<10	<10
	2015	10	<10	13	<10	<10	<10
Wheatfeed	2012	7	<10	28	<10	<10	<10
	2013	0	<10	<10	<10	<10	<10
	2014	0	<10	<10	<10	<10	<10
	2015	10	14	<10	<10	<10	<10
Feed barley	2012	0	<5	<5	<5	<5	<5
	2013	33	12	41	<5	<5	<5
	2014	18	<5	43	<5	<5	<5
	2015	18	59	881	<5	<5	<5
Feed oats	2012	83	139	1030	<5	62	<5
	2013	77	158	899	<5	68	<5
	2014	92	175	905	<5	54	N/A
	2015	73	256	983	<5	185	<5
Oatfeed	2012	100	580	1296	175	420	N/A
	2013	100	1226	3285	64	855	N/A
	2014	100	1197	4310	148	768	N/A
	2015	92	1421	2582	<5	1230	N/A

Other trichothecenes

Other than nivalenol, the other trichothecenes, 3-acetyl-DON, 15-acetyl-DON, diacetoxyscirpenol, fusarenone-X and neosolaniol, were rarely detected in any samples.

Field mycotoxins: Fusarium toxin zearalenone (ZON)

Zearalenone was analysed in all freshly harvested samples of food and feed grain (Tables 15, 16 and 18). Incidence, mean and maximum levels in milling wheat were higher than malting barley for the corresponding harvest seasons. The highest level of ZON in milling wheat was determined in a sample from the 2012 harvest. The level determined, 234 µg/kg, exceeded the maximum limit of 100 µg/kg. Incidence, mean and maximum levels for milling wheat declined from 2012 to 2015 harvests. 2012 harvest samples were also responsible for the highest levels of ZON in feed wheat, wheatfeed, feed barley and feed oats.

Mean and maximum levels in oatfeed were generally low even when the level of incidence was 100%.

No correlation could be drawn from ZON levels in the paired barley and malt samples (Table 17) due to the extremely low levels of incidence in three of the harvest periods (2013-15). Levels and incidence in 2012 paired samples were generally similar.

Table 15: Zearalenone in freshly harvested milling wheat

Harvest year	Incidence	Mean	Maximum	Minimum	Median	Mode
	%	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
2012	63	16	234	<2	16	<2
2013	62	6	65	<2	3	<2
2014	15	<2	10	<2	<2	<2
2015	8	<2	8	<2	<2	<2

Table 16: Zearalenone in freshly harvested malting barley

Harvest year	Incidence	Mean	Maximum	Minimum	Median	Mode
	%	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
2012	30	6	42	<2	<2	<2
2013	5	2	10	<2	<2	<2
2014	7	<2	24	<2	<2	<2
2015	0	<2	<2	<2	<2	<2

Table 17: Zearalenone in malting barley and malt pairs

Harvest year	Sample	Incidence	Mean	Maximum	Minimum	Median	Mode
		%	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
2012	Barley	42	4	29	<2	<2	<2
2012	Malt	53	5	35	<2	2	<2
2013	Barley	0	<2	<2	<2	<2	<2
2013	Malt	14	2	18	<2	<2	<2
2014	Barley	0	<2	<2	<2	<2	<2
2014	Malt	0	<2	<2	<2	<2	<2
2015	Barley	0	<2	<2	<2	<2	<2
2015	Malt	0	<2	<2	<2	<2	<2

Table 18: Zearalenone in cereal feedingstuffs

Sample	Harvest Year	Incidence	Mean	Maximum	Minimum	Median	Mode
		%	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
Feed Wheat	2012	89	46	118	<2	34	N/A
	2013	56	7	37	<2	2	<2
	2014	30	3	18	<2	<2	<2
	2015	30	<2	4	<2	<2	<2
Wheatfeed	2012	100	66	316	3	52	N/A
	2013	100	31	173	7	23	14
	2014	90	7	26	<2	4	2
	2015	60	4	20	<2	<2	<2
Feed barley	2012	33	29	113	<2	<2	<2
	2013	33	3	11	<2	<2	<2
	2014	0	<2	<2	<2	<2	<2
	2015	0	<2	<2	<2	<2	<2
Feed oats	2012	67	14	106	<2	4	<2
	2013	0	<2	<2	<2	<2	<2
	2014	8	2	24	<2	<2	<2
	2015	0	<2	<2	<2	<2	<2
Oatfeed	2012	100	16	30	5	14	N/A
	2013	43	7	37	<2	<2	<2
	2014	30	6	25	<2	<2	<2
	2015	0	<2	<2	<2	<2	<2

4.2. Ergot alkaloids

Ergot alkaloids are mycotoxins produced by the fungi of the *Claviceps* genus, the most notable in Europe being *Claviceps purpurea*. The fungal structure known as ergot, or sclerotium replaces the developing grain or seed of the host. The most prominent ergot alkaloids produced by the *Claviceps* species which are contained in the sclerotia are ergotmetrine, ergosine, ergotamine, ergocristine, ergocryptine and ergocornine, and their corresponding –inines.

In June 2012, following a request from the European Commission, EFSA's Panel on Contaminants in the Food Chain delivered a scientific opinion on the risks to human and animal health to ergot alkaloids in food and feed, and requested that Member States to gather data on the presence of ergot alkaloids in relevant food and feed commodities.

The Commission requested that data on the presence of ergot alkaloids in cereals and cereal products by 30th September 2016 in order to allow setting appropriate and achievable maximum levels to provide a high level of human health protection. The principal ergot alkaloids in the four harvest periods that were either the most frequently detected or were determined at the highest level, were ergotamine, ergosine and ergocristine. In each case, these were usually accompanied by lower levels of the corresponding epimers. There is considerable variation of principal alkaloids from season to season and between sample types. Incidences of multiple ergot alkaloids (and epimers) were generally more common than single alkaloids in all sample types analysed. In several instances, all six alkaloids and epimers were determined in the same sample. Wheatfeed and oatfeed were the most frequently contaminated sample types. The highest calculated median values were in the wheatfeed samples followed by oatfeed.

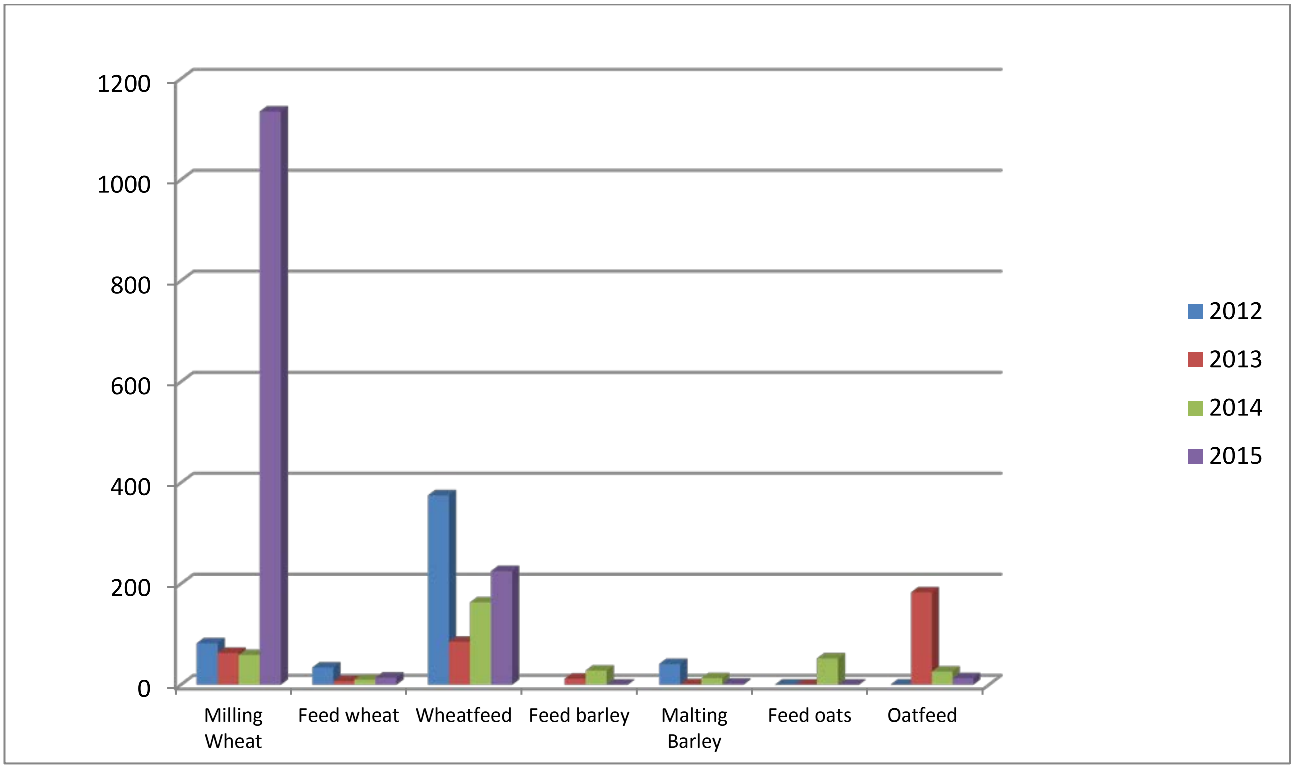
The highest total alkaloid content was 78679 µg/kg, which was determined in a 2015 harvest milling wheat sample imported from Germany. No further information on this sample was available. Other than in wheatfeed and oatfeed, the median values for the vast majority of samples were below the method LOQ.

Table 19: Ergot alkaloids harvest samples – Total combined ergot alkaloid values

Sample	Harvest Year	Incidence	Mean	Maximum	Minimum	Median	Mode
		%	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
Milling Wheat	2012	25	68	1364	0	0	0
	2013	49	63	1381	0	0	0
	2014	45	59	1738	0	0	0
	2015	41	1135	78679	0	0	0
Feed wheat	2012	38	34	163	0	0	0
	2013	50	7	25	0	1	0
	2014	20	10	90	0	0	0
	2015	30	14	70	0	0	0
Wheatfeed	2012	92	532	2206	0	170	N/A
	2013	94	85	249	0	67	19
	2014	100	163	811	3	112	N/A
	2015	100	225	542	6	205	N/A
Feed barley	2012	14	10	70	0	0	0
	2013	22	12	106	0	0	0
	2014	9	28	309	0	0	0
	2015	6	0	1	0	0	0

Malting Barley	2012	7	3	40	0	0	0
	2013	13	1	23	0	0	0
	2014	29	13	149	0	0	0
	2015	53	2	27	0	1	0
Feed oats	2012	0	0	0	0	0	0
	2013	0	0	0	0	0	0
	2014	25	52	601	0	0	0
	2015	0	0	0	0	0	0
Oatfeed	2012	70	181	1090	0	48	0
	2013	57	6	20	0	5	0
	2014	70	26	121	0	10	0
	2015	85	13	54	0	8	2

Figure 3: Mean value (μg/kg) of total Ergot Alkaloids in all sample types 2012-15



4.3. Ochratoxin A

Milling wheat samples taken from grain stores in January and March following the 2012, 2013, 2014 and 2015 harvests were tested for Ochratoxin A. Results are shown in Table 20.

Table 20: Ochratoxin A in stored milling wheat

Harvest year	Sampling month	Incidence	Mean	Maximum	Minimum	Median	Mode
		%	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
2012	January 2013	6	<0.1	0.8	<0.1	<0.1	<0.1
2012	March 2013	18	0.3	9.1	<0.1	<0.1	<0.1
2013	January 2014	18	1.0	41.7	<0.1	<0.1	<0.1
2013	March 2014	10	0.2	3.9	<0.1	<0.1	<0.1
2014	January 2015	16	0.3	3.6	<0.1	<0.1	<0.1
2014	March 2015	10	0.3	9.9	<0.1	<0.1	<0.1
2015	January 2016	6	0.2	4.6	<0.1	<0.1	<0.1
2015	March 2016	8	<0.1	1.2	<0.1	<0.1	<0.1

The incidence of contamination was low. Less than 20% of samples in each harvest period contained detectable levels above the limit of quantification (> 0.1µg/kg) of ochratoxin A. Mean levels were low and generally just above the limit of quantification. Three samples were above the EU maximum of 5 µg/kg. In these instances, the mills were advised immediately and action taken by the companies that supplied the samples.. The data collected did not suggest that extended storage led to higher incidence or levels of Ochratoxin A. Median levels were all below limit of quantification.

Other than in the 2012 set, malting barley samples showed slightly lower levels of incidence and lower mean levels than the wheat samples (Table 21). One sample was above the EU maximum level of 5 µg/kg. Data from the 2012 sample set may be skewed due to the incidence of this sample (11.5 µg/kg). When these barleys were then used to produce malts, there was a slight increase in incidence of detectable ochratoxin A. Other than 2012 data, mean and maximum values in each of the three years remained largely the same, whilst median levels were all below limit of quantification

Table 21: Ochratoxin A in stored barley and malt pairs

Harvest year	Sample	Incidence	Mean	Maximum	Minimum	Median	Mode
		%	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
2012	Barley	36	1.0	11.5	<0.1	<0.1	<0.1
2012	Malt	52	0.2	1.5	<0.1	0.1	<0.1
2013	Barley	0	<0.1	<0.1	<0.1	<0.1	<0.1
2013	Malt	9	<0.1	0.5	<0.1	<0.1	<0.1
2014	Barley	5	<0.1	0.4	<0.1	<0.1	<0.1
2014	Malt	16	<0.1	0.4	<0.1	<0.1	<0.1
2015	Barley	5	<0.1	0.5	<0.1	<0.1	<0.1
2015	Malt	20	0.2	0.8	<0.1	<0.1	<0.1

Although it has been shown that Ochratoxin A can either be formed (unsuitable conditions) or removed (steeping stages) during malting, the difficulty and large potential error in effectively sampling large bulks of grain to obtain a reliable representative sample effectively makes it impossible to draw any conclusion between levels in individual pairs of barley and malt. The incidence of Ochratoxin A was significantly higher in feed than in food grains. The highest levels of incidence were in wheatfeed and oatfeed samples (Table 22). Mean values in all sample types were below 2 µg/kg. The highest concentration (139 µg/kg) was found in a sample of feed oats.

Table 22: Ochratoxin A in stored feed cereals

Sample	Harvest Year	Incidence	Mean	Maximum	Minimum	Median	Mode
		%	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
Feed Wheat	2012	13	0.2	1.6	<0.2	<0.2	<0.2
	2013	11	0.2	2.8	<0.1	<0.1	<0.1
	2014	20	0.3	5.5	<0.1	<0.1	<0.1
	2015	14	0.2	4.9	<0.1	<0.1	<0.1
Wheatfeed	2012	89	0.6	1.8	<0.2	0.3	0.2
	2013	70	0.4	0.7	<0.1	0.4	0.05,0.5
	2014	80	0.5	1.6	<0.1	0.5	<0.1
	2015	100	0.7	1.3	0.3	0.7	0.7
Feed barley	2012	17	0.2	1.7	<0.1	<0.1	<0.1
	2013	33	0.5	9.1	<0.1	<0.1	<0.1
	2014	17	1.4	29.5	<0.1	<0.1	<0.1
	2015	19	0.6	6.1	<0.1	<0.1	<0.1
Feed oats	2012	45	1.3	3.0	<0.1	<0.1	<0.1
	2013	60	1.4	17.0	<0.1	<0.1	<0.1
	2014	27	12.8	139	<0.1	<0.1	<0.1
	2015	20	1.5	14.5	<0.1	<0.1	<0.1
Oatfeed	2012	88	1.3	3.6	<0.1	<0.1	<0.1
	2013	50	0.7	1.8	<0.1	0.6	<0.1
	2014	88	1.1	1.9	<0.1	1.1	0.5
	2015	75	0.8	2.0	<0.1	0.7	<0.1

4.4. **Pesticide Residues**

Storage insecticides and selected field fungicides

Stored samples were tested for a wide range of pesticides including organochlorines, organophosphates, carbamates and synthetic pyrethroids. All samples selected for analysis were analysed for the same suite of pesticides. The residues sought are listed in the Materials and Methods section (Table 5). Milling wheat and feed grains were sampled six to eight months after harvest and malting barleys three to eight months after harvest. Most stored grain samples are free from detectable pesticide residues. All levels of permitted insecticides were below respective EU MRLs.

Over the four year sampling period, only eight different pesticides were detected in any of the samples analysed and in virtually all cases the levels were very low. The organophosphates pirimiphos-methyl and chlorpyriphos methyl were by far the most commonly detected residues in 2012 food grain harvest samples. However, from the 2013 harvest onwards, the use of the synthetic pyrethroids, deltamethrin followed by cypermethrin were more frequently detected. In 2015, harvested food grain samples these were the most commonly determined residues, although all residues were well below respective MRLs.

Malathion was detected in a small percentage of wheat samples. Malathion is not currently approved for use in the UK but all three of the samples where it was found were imported. The extremely low levels suggest it was treated some time prior to import. All three samples were significantly below the current MRL. One sample of stored milling wheat from the 2013 harvest contained chlorpropham above the EU MRL of 0.02 mg/kg. The presence of this residue was due to cross contamination and not incorrect application of this agrochemical.

Table 23: Incidence (%) of pesticide residues in stored grain

Sample	Harvest Year	Incidence (%) of samples containing pesticide residues > LOD	Incidence (%) of samples containing single pesticide residues > LOD	Incidence (%) of samples containing multiple pesticide residues > LOD
Milling wheat	2012	6	6	0
	2013	14	10	4
	2014	18	14	4
	2015	28	24	4
Malting barley	2012	5	0	5
	2013	27	27	0
	2014	11	11	0
	2015	5	5	0
Feed wheat	2012	19	19	0
	2013	3	3	0
	2014	0	0	0
	2015	0	0	0
Wheatfeed	2012	56	44	11
	2013	60	50	10
	2014	11	11	0
	2015	22	11	11
Feed barley	2013	31	23	8
	2014	0	0	0
	2015	0	0	0
Oatfeed	2013	50	50	0
	2014	38	13	25
	2015	25	25	0

Table 24: Pesticide residues detected in stored grain

Sample	Harvest Year	Pesticides determined. (Residues > LOD) %	Maximum level determined mg/kg
Milling wheat	2012	Pirimiphos methyl (6%)	0.42
	2013	Pirimiphos methyl (6%)	0.20
		Chlorpyrifos methyl (4%)	*0.02*
		Deltametthrin (2%)	*0.05*
		Malathion (2%)	0.05
		Chlorpropham (4%)	0.23
	2014	Chlorpyrifos methyl (6%)	0.24
		Pirimiphos methyl (4%)	1.08
		Malathion (2%)	0.03
		Deltamethrin (10%)	0.07
	2015	Chlorpyrifos methyl (4%)	0.02
		Pirimiphos methyl (6%)	0.38
		Malathion (2%)	0.03
		Cypermethrin (6%)	0.02
		Deltamethrin (14%)	0.23
Malting barley	2012	Pirimiphos-Methyl (5%)	0.01
		Chlorpyriphos-methyl (5%)	0.05
	2013	Pirimiphos-Methyl (5%)	0.05
		Chlorpyriphos-methyl (5%)	0.10
		Deltamethrin (18%)	0.95
	2014	Pirimiphos-Methyl (5%)	0.01
		Deltamethrin (5%)	0.03
	2015	Deltamethrin (5%)	0.07
Feed wheat	2012	Pirimiphos-Methyl (14%)	0.13
		Chlorpyriphos-methyl (5%)	0.99
	2013	Tebuconazole (3%)	0.03
	2014	-	
	2015	-	
Wheatfeed	2012	Pirimiphos-Methyl (56%)	0.40
		Chlorpyriphos-methyl (11%)	0.04
	2013	Pirimiphos-Methyl (60%)	0.38
		Chlorpyriphos-methyl (10%)	0.58
	2014	Pirimiphos-Methyl (11%)	1.44
	2015	Chlorpyrifos methyl (22%)	0.14
		Pimimiphos methyl (11%)	0.79
Feed barley	2013	Pirimiphos-Methyl (31%)	0.10
		Chlorpyrifos methyl (8%)	0.01
	2014	-	
	2015	-	
Oatfeed	2013	Pirimiphos-Methyl (50%)	0.09
	2014	Pirimiphos-Methyl (38%)	0.04
		Chlorpyrifos methyl (25%)	0.02
	2015	Chlorpyriphos methyl (13%)	0.01
		Deltamethrin (13%)	0.01

Glyphosate

Glyphosate is a herbicide that is also authorised for use as a desiccant on cereals, where it may be used immediately before harvesting. It is one of the residues most frequently reported in official surveys of cereals in the UK. In the 2015 PRiF wheat monitoring programme, it was detected in 33% of samples analysed. All residues, however, were significantly below the MRL of 10 mg/kg. Selected samples of barley and wheat from the 2012 to 2015 harvests were analysed for glyphosate. Results are shown in Table 25 and 26. The levels and incidence in milling wheat were largely in line with the 2015 PRiP survey results with all samples being well below the MRL. Incidence of glyphosate in malting barley was slightly higher than in milling wheat, but again all results were well below the MRL.

Table 25: Glyphosate in freshly harvested milling wheat

Harvest year	Incidence	Mean	Maximum	Minimum	Median	Mode
	%	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
2013	40	284	2236	<50	<50	<50
2014	40	154	1403	<50	<50	<50
2015	30	122	924	<50	<50	<50

Table 26: Glyphosate in freshly harvested malting barley

Harvest year	Incidence	Mean	Maximum	Minimum	Median	Mode
	%	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
2012	72	1010	3200	<50	490	<50

* Mean is calculated by assuming that all samples below the limit of detection contained half that limit.

Plant growth regulators

Milling wheat samples were tested for both chlormequat and mepiquat in each of the four harvests. (Tables 27 and 28). Malting barley samples from the 2012 harvest were also analysed. Chlormequat was detected in the majority of milling wheat samples (≥90% of samples analysed). These results are in line with the values determined in the 2015 PRiP wheat monitoring programme. A slightly lower level of incidence of chlormequat was determined in malting barley. Although the incidence of residues was high, concentrations were well below the MRL (2 mg/kg). Levels and incidence of mepiquat were much less common which was in line with previous data.

Table 27: Chlormequat in freshly harvested milling wheat

Harvest year	Incidence	Mean	Maximum	Minimum	Median	Mode
	%	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
2012	90	240	468	<10	222	90
2013	95	291	1059	<10	255	N/A
2014	95	197	519	<10	177	87
2015	100	218	885	21	141	N/A

Table 28: Mepiquat in freshly harvested milling wheat

Harvest year	Incidence	Mean	Maximum	Minimum	Median	Mode
	%	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
2012	15	11	66	<10	<10	<10
2013	10	<10	46	<10	<10	<10
2014	10	<10	81	<10	<10	<10
2015	0	<10	<10	<10	<10	<10

Table 29: Chlormequat in freshly harvested malting barley

Harvest year	Incidence	Mean	Maximum	Minimum	Median	Mode
	%	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
2012	67	120	510	<10	60	<10

Table 30: Mepiquat in freshly harvested malting barley

Harvest year	Incidence	Mean	Maximum	Minimum	Median	Mode
	%	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
2012	22	50	660	<10	<10	<10

4.5 Elements

Selected freshly harvested samples from the 2012, 2014 and 2015 harvests were analysed for a range of elements including, cadmium, lead, arsenic, mercury, copper and nickel. Results are summarised in Tables 31 to 41.

In 2014, the European Commission published Recommendation 2014/193/EU on the reduction of the presence of cadmium in foodstuffs. Maximum levels of cadmium will be reviewed in 2018/19 with a view to reducing them. Cadmium levels in milling wheat and malting barley were all within the current EU limits (0.2 mg/kg for wheat; 0.1 for barley). Maximum levels in milling wheat reduced from 2012 (0.14 mg/kg) to 2015 (0.07mgkg) harvests.

Table 31: Cadmium in selected freshly harvested grain samples

Sample	Harvest Year	Incidence	Mean	Maximum	Minimum	Median	Mode
		%	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
Milling Wheat	2012	100	0.05	0.14	0.02	0.04	0.04
	2014	100	0.03	0.11	0.01	0.03	0.03
	2015	100	0.04	0.07	0.01	0.03	0.03
Feed Wheat	2014	100	0.04	0.06	0.02	0.03	0.03
Wheatfeed	2014	100	0.07	0.09	0.05	0.07	0.09
Feed barley	2012	80	0.01	0.01	<0.01	0.01	0.01
Feed oats	2012	89	0.03	0.05	<0.01	0.02	0.01,0.02,0.05
Oatfeed	2012	100	0.01	0.03	0.01	0.01	0.01
Malting Barley	2014	92	0.01	0.02	<0.01	0.01	0.01
	2015	100	0.01	0.03	0.01	0.01	0.01

Lead levels were very similar to those determined in previous projects. All levels were below current EU limits.

Table 32: Lead in selected freshly harvested grain samples

Sample	Harvest Year	Incidence	Mean	Maximum	Minimum	Median	Mode
		%	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
Milling Wheat	2012	80	0.01	0.04	<0.01	0.01	0.01
	2014	27	<0.01	0.06	<0.01	<0.01	<0.01
	2015	10	<0.01	0.01	<0.01	<0.01	<0.01
Feed Wheat	2014	33	0.01	0.03	<0.01	<0.01	<0.01
Wheatfeed	2014	89	0.04	0.18	<0.01	0.03	0.03
Feed barley	2012	100	0.03	0.04	0.01	0.03	0.03
Feed oats	2012	78	0.03	0.18	<0.01	0.01	0.01
Oatfeed	2012	88	0.11	0.41	<0.01	0.06	0.06
Malting Barley	2014	50	0.01	0.04	<0.01	<0.01	<0.01
	2015	78	0.01	0.03	<0.01	<0.01	<0.01

The European Commission has recommended that Member States monitor the presence of arsenic in foods- including cereal grains and cereal based products during 2016, 2017 and 2018. In addition to inorganic and total arsenic, they recommended that speciation data also be collected. Mean and maximum levels were similar to those collected in project RD-2008-3572).

Table 33: Arsenic in selected freshly harvested grain samples

Sample	Harvest Year	Incidence	Mean	Maximum	Minimum	Median	Mode
		%	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
Milling Wheat	2012	84	0.02	0.05	<0.01	0.02	0.01
	2014	4	<0.01	0.02	<0.01	<0.01	<0.01
	2015	35	<0.01	0.01	<0.01	<0.01	<0.01
Feed Wheat	2014	0	<0.01	<0.01	<0.01	<0.01	<0.01
Wheatfeed	2014	68	0.01	0.03	<0.01	0.01	0.02
Feed barley	2012	100	0.03	0.05	0.01	0.02	0.02
Feed oats	2012	89	0.02	0.10	<0.01	0.02	0.02
Oatfeed	2012	100	0.04	0.10	0.02	0.03	0.03
Malting Barley	2014	38	0.01	0.05	<0.01	<0.01	<0.01
	2015	89	0.01	0.02	<0.01	0.01	0.01

Mercury was not detected in the majority of sample types (limit of detection was 0.01 mg/kg). The only detectable levels were in 2014 harvest milling wheat samples.

Table 34: Mercury in selected freshly harvested grain samples

Sample	Harvest Year	Incidence	Mean	Maximum	Minimum	Median	Mode
		%	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
Milling Wheat	2012	0	<0.01	<0.01	<0.01	<0.01	<0.01
	2014	7	<0.01	0.04	<0.01	<0.01	<0.01
	2015	0	<0.01	<0.01	<0.01	<0.01	<0.01
Feed Wheat	2014	0	<0.01	<0.01	<0.01	<0.01	<0.01
Wheatfeed	2014	0	<0.01	<0.01	<0.01	<0.01	<0.01
Feed barley	2012	0	<0.01	<0.01	<0.01	<0.01	<0.01
Feed oats	2012	0	<0.01	<0.01	<0.01	<0.01	<0.01
Oatfeed	2012	0	<0.01	<0.01	<0.01	<0.01	<0.01
Malting Barley	2014	0	<0.01	<0.01	<0.01	<0.01	<0.01
	2015	0	<0.01	<0.01	<0.01	<0.01	<0.01

Table 35: Copper in selected freshly harvested grain samples

Sample	Harvest Year	Incidence	Mean	Maximum	Minimum	Median	Mode
		%	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
Milling Wheat	2012	100	2.91	4.13	1.40	2.91	N/A
Feed barley	2012	100	2.53	3.56	1.79	2.38	N/A
Feed oats	2012	100	3.25	3.81	2.66	3.26	N/A
Oatfeed	2012	100	2.24	2.85	1.93	2.21	N/A

In response from an EFSA request to monitor nickel levels in foods, including cereals and cereal products, selected 2014 and 2015 harvest samples were included within the 2014-2016 surveillance programme.

Table 36: Nickel in selected freshly harvested grain samples

Sample	Harvest Year	Incidence	Mean	Maximum	Minimum	Median	Mode
		%	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
Milling Wheat	2014	100	0.10	0.31	0.02	0.09	0.09
	2015	100	0.13	0.24	0.05	0.12	0.12
Feed Wheat	2014	89	0.08	0.18	<0.02	0.06	0.06,0.10
Wheatfeed	2014	100	0.31	0.50	0.13	0.31	0.31
Malting Barley	2014	100	0.15	0.27	0.08	0.15	0.14
	2015	100	0.09	0.13	0.06	0.09	0.08

4.6 Dioxins

Dioxins had not been incorporated in previous surveillance programmes. In 2008 (Ireland) and 2011 (Germany), incidences of dioxin contamination were reported in animal feed. Although the dioxin contamination was not attributed to cereal sources it did identify a gap in the range of analytes surveyed.

In this programme, a small selection of feed samples from the 2014 and 2015 harvests were included within the programme. The results were in line with those generated from a larger AIC dioxin surveillance programme and below the regulatory limit of 1.5 ng/kg.

Table 37: Dioxins in selected feed samples samples

Sample	Harvest Year		Lower Bound@ 0% moisture –ng/kg (minimum value)	Upper Bound@ 0% moisture –ng/kg (minimum value)
Feed Wheat	2014	Dioxins and Furans (Based on US EPA 1013)	0.0	0.17
Feed Barley	2014	Dioxins and Furans (Based on US EPA 1013)	0.0	0.14
Feed Wheat	2015	Dioxins and Furans (Based on US EPA 1013)	0.0	0.21

4.7. **Chlorates**

Chlorate is a by-product of chlorine based water treatment and can be present as an impurity in water based disinfectants. Chlorates in food may arise from the use of chlorinated water in processing techniques or from the cross contamination of chlorinated disinfected processing equipment. Revised MRLs for chlorate are currently under discussion.

Table 38: Chlorate in malting barley and malt

Sample	Harvest Year	Incidence	Mean	Maximum	Minimum	Median	Mode
		%	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
Barley	2015	0	<10	<10	<10	<10	<10
Malt	2015	20	20	100	<10	<10	<10

5 Discussion

Overall, the data established by this project suggest that the bulk of UK-grown cereals comply with EU and UK legislation and recommendations with regard to the presence of contaminants.

Mycotoxins: The trichothecene, deoxynivanol was regularly detected on food and feed grains surveyed. Concentrations for the vast majority of samples analysed over the four-year surveillance period were well below maximum limits. Five samples in the 2012 harvest exceeded the maximum limit. However, this represented less than 1% of the total samples analysed. Deoxynivalenol was the most frequently detected trichothecene in wheat and barley, whilst HT-2 and T-2 were the most common in oats.

Ochratoxin A was detected less frequently in food grains and concentrations were generally very low with mean values just over than the reporting limit of the method (0.1 µg/kg), suggesting that toxin synthesis in food grains is being successfully kept in check by storage conditions. Occasional samples exceeded legal limits, but as in previous years, due to sampling difficulties, these levels were not replicated upon re-sampling. The well-recognised difficulties with obtaining representative samples remain a problem. Incidence in compounded samples was similar to the previous study, in that wheatfeed, and oatfeed concentrations were significantly higher but levels were well below guideline levels.

Pesticides: Chlormequat and glyphosate, although regularly the most detected agrochemicals, were always determined at levels that werewell below their respective MRLs. There would appear to be a shift in the type of post-harvest insecticides being applied to food grains. In previous projects, and in the first two years of this surveillance programme, two organophosphates, pirimiphos methyl and chlorpyrifos methyl were the most frequently detected insecticide residues. In the 2014 and 2015 harvests, two synthetic pyrethroids, deltamethrin and cypermethrin have now replaced them as the most commonly detected insecticides.

Levels, however, were generally very low and in the vast majority of cases, only marginally above the detection limit and all levels were below respective MRLs. This data demonstrates correct application of agrochemicals and following of Good Agricultural Practice.

Heavy metals: Concentrations of heavy metals were generally low in the samples tested and well below current legal limits. In response from an EFSA request to monitor nickel levels in foods, including cereals and cereal products, selected 2014 and 2015 harvest samples were included within the 2014-16 surveillance programme.

Chlorate: Chlorate is no longer approved as a pesticide according to Commission Decision No.2008/865/EC. Consequently it has a default MRL of 0.01 mg/kg.
The CONTAM Panel has recommended that more information about the impact of food processing on chlorate residues in food be collected. This is an ongoing issue and is continuing to be discussed by the European Commission Standing Committee on Plants Animals Food and Feed (PAFF) –Residues Section. Revised MRLs for chlorate are currently under discussion.

Dioxins: Dioxins had not been incorporated in previous surveillance programmes. In 2008 (Ireland) and 2011 (Germany), incidences of dioxin contamination were reported in animal feed. Although the dioxin contamination was not attributed to cereal sources, it did identify a gap in the range of analytes surveyed.

In this programme, a small selection of feed samples from the 2014 and 2015 harvests were included within the programme. The results were in line with those generated from a larger AIC dioxin surveillance programme and below the regulatory limit of1.5 ng/kg.

Emerging issues:
Proposed changes to MRL levels for deltamethrin, potential changes to the residue definition of chlormequat, the setting of chlorate MRLs, lowering cadmium maximum levels and the setting of maximum levels for ergot alkaloids need to be monitored in order to assess the potential future impact on residue compliance.

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