## HGCA Grain sampling guide



# The value of grain sampli

## Introduction

Understanding the quality and condition of grain is crucial. Accurate sampling at each stage of the grain chain is required to develop that understanding. It should help to reduce waste and minimise charges, claims and rejections.

This guide brings together the key requirements for effective grain sampling for everyone involved, from growing to purchasing. It seeks to minimise duplication of effort, maximising efficiency. In this guide, sampling refers to the collection of physical grain and also sampling for moisture, temperature, pests and moulds.

For many years, sampling grain has been important in measuring key quality parameters in combinable crops (eg Hagberg Falling Number, nitrogen content and specific weight). In recent years, however, other challenges (including mycotoxins) have emerged, requiring the industry to demonstrate due diligence; samples of grain traded are part of that evidence. Grain sampling is, therefore, even more important and must be undertaken using appropriate methods at the most relevant points along the grain chain.

#### This guide has been endorsed by:



## Grain marketing

#### Before harvest

- Select appropriate varieties for the intended market
- Assess ability to segregate and store different varieties and qualities at harvest – is this sufficient to support the number of varieties grown?
- Before marketing, understand your contractual obligations and rights in relation to sampling
- When marketing quality grain, consider managing the feed base price and premium separately – the majority of price volatility results from the feed base price

#### After harvest

- Maintain communication with buyers on quality and variation so that grain can be allocated to the most appropriate user
- Where grain is sampled more than once on-farm, do not necessarily rely on the highest quality analysis results because variation needs to be considered – any 'heap' of grain will have variation within it
- Take and retain a representative sample from each lorry loaded – these can support decision-making if a dispute arises

For more information, see HGCA's Cereal/Oilseed Sellers' Checklists and ensure anyone involved in selling combinable crops is familiar with the AIC No. 1 and FOSFA 26A contracts.

Best practice sampling at each stage of the grain chain
<b>1. Know the harvested quality:</b> At harvest, analysis can only determine grain quality if sampling is representative of grain loads coming into store. Results from analysis of harvested grain will confirm if grain meets the proposed market's criteria.
Targets for moisture and temperature of stored grain are set out in the HGCA Grain storage guide (www.hgca.com/grainstorage). Sampling and analysis at harvest indicate the action needed and timescale to meet those targets.
2. Protect the harvested quality: During storage, sampling for temperature and moisture content is required to assess changes in physical condition and to verify that storage targets are met. Without effective drying and cooling, spoilage may occur and potential market opportunities may be lost.
2. Know what look as the starray Armithan is a starray with a starray starray in the starray starray is a starray of the starray starray is a starray starray in the starray starr
3. Know what leaves the store: At outloading from the store, representative samples taken as lorries are loaded provide evidence of what has been dispatched. This is the best opportunity for the grower's and purchaser's assessments of delivered quality to match.
4. Know the quality received: At receipt, most buyers (merchants or processors) will sample from each delivery lorry, often using automated equipment, and should follow the best practice recommended by assurance schemes, such as TASCC.



## ng

Monitor for

moisture,

temperature

and pests.

Take at least 10 samples as each lorry is loaded, to provide two 1kg representative samples.

Take at least 8 samples from each lorry, totalling at least 3kg. Mix and subsample.

#### An example of sampling opportunities Take Take samples Combine and mix to samples and assess produce an moisture aggregate content. sample representing no more than 100t. Tipping Analyse a trailer representative sample for market criteria. Drier/ cleaner

Store

Lorry

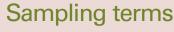
Processor

samples taken from ex-harvest aggregate samples or from on-farm stores for indication of marketability.

Group

storage

Merchant:



**Incremental sample** – any single sample taken by spear, jug or other means, to be combined with others.

Aggregate sample – a large sample comprising all smaller samples (ie incremental samples) taken at one point in the grain chain.

**Representative sample** – a final quantity of grain from the aggregate sample using appropriate mixing/sampling procedures (see page 17). This sample is suitable for laboratory analysis.

Contents	Page
Preparation	4
Why is sampling important?	6
Sampling at harvest	8
Sampling and monitoring in sto	re 10
Sampling at outloading	12
Sampling at commercial intakes	14
Sampling and mycotoxins	16
Mixing and sub-sampling	17
Further information	18

The value of grain sampling

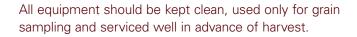
# Preparation

## Equipment

Ahead of harvest, ensure that all equipment is ready for use.

Equipment for sampling should include:

- Sampling spear
- Measuring jugs
- Sample bags capable of holding samples of 1kg of grain
- Sealed containers to hold samples of 5-10kg of grain
- Labels that are suitable to be applied to bags and containers (if bags do not have white printed writing strips)
- Indelible pens standard ballpoint ink can fade in a matter of months
- Moisture meter ensure it is serviced and calibrated well ahead of harvest
- Record sheets
- Temperature probes consider using automated systems
- Insect traps



Containers must be capable of being sealed, to:

- minimise moisture loss
- prevent rodent access
- avoid contamination by dust and other grain



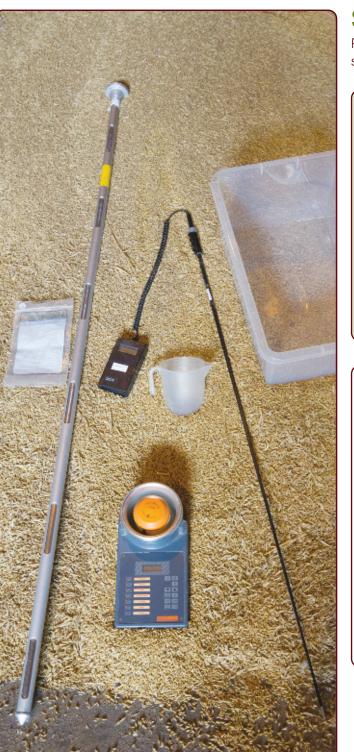
Container for gathering incremental samples











The pieces of equipment shown are examples and no recommendation of a specific model is implied

## Sample storage

Prepare a cool, dry area that is rodent-free to store grain samples as required.

For more information, see Rodent control in agriculture – an HGCA guide.

Rodent control in agriculture – an HGCA guide Second edition



## General safety information

- Ensure training, where appropriate, is given and all those involved in sampling are aware of the risk areas
- Ensure all equipment is suitable for purpose and has been checked to ensure fitness for use
- Ensure staff and visitors are aware of the vehicular movements in and around the loading/unloading areas
- Always ensure people sampling from bulk grain stores are accompanied
- No one should enter a closed silo



# Why is sampling importa

## Sources of variation

## Variation in growing grain

Grain is extremely variable. Variation not only occurs across a farm or field but even within a plant and within individual ears. The causes of variation can include soil types, local nutrient availability, different varieties or sowing dates, hedge and boundary effects and late tillering.

## Variation in harvested grain

Timeliness of harvest and prevailing harvest weather affect grain quality. During a harvest day, grain moisture content changes and trailer loads will differ due to natural variation in each harvested field.

Diseases may have affected only some parts of a field and possibly at a low level of incidence. This may or may not have led to potential contamination by, for example, mycotoxins.

## Variation between undried and dried grain

Considerable variation, particularly in temperature and moisture content, can occur within newly harvested grain stored on-floor before being conditioned.

When ambient or near-ambient air is used to dry bulk grain, a 'drying front' moves up through the grain.

Grain stored after high temperature drying will vary less in moisture but temperatures will generally be higher, requiring more urgent cooling effort.

Excessive heat generated within grain stored wet, or applied during high temperature drying, can damage grain protein and germination. Over-drying rapeseed can make it brittle and subject to a rapid increase in free fatty acids during storage. Certain characteristics are not affected, such as nitrogen content.

## Variation within the farm store

Segregating grain into identifiable lots of similar quality maximises the likelihood of achieving market specification. Identifying grain in relation to samples taken at harvest is easier in smaller storage units, for instance, in discrete bins up to 60t capacity.

Modern storage methods, however, often involve large bulks of grain stored at depths of up to 6–8m 'pushed-up' by specialist equipment. This means each trailer load will be moved up the grain face after tipping on the floor. This mixing is compounded as grain moves back down the slope during outloading. There is little chance of identifying individual trailer loads, or even sets of trailer loads, within such a bulk.

## Action:

- Keep varieties separate
- For each variety, identify areas likely to produce grain of significantly different quality (eg headlands or different fields) and keep grain from these areas separate, wherever possible

#### Action:

- Every trailer load should be sampled for moisture content
- Records should reflect where grain within the store came from to enable segregation by quality – this is required practice under most assurance schemes
- Segregate grain lots that may be more affected by fungal disease or have quite different specific weights

#### Action:

- Sample after batch or continuous flow drying to assess quality for markets
- With ambient or near-ambient drying, moisture monitoring is important to confirm that the drying front has completed its passage through the bulk
- Seek to treat similar batches of grain in the same way
- Ensure drier settings are appropriate for the commodity being dried – for example, do not use feed wheat settings for drying malting barley
- Aim to use the cooling section of the hot-air drier to best effect and cool grain quickly once it is in long-term storage

#### Action:

- Aim to store in units of around 100t, in identifiable units where possible
- Where grain is loaded out from large bulk stores, samples taken during lorry loading provide critical records of what has been dispatched
- Do not rely on the analytical results reported on grain samples drawn at harvest intake, other than as an indication of marketing potential and the need for taking action in the store

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## Variation within loads delivered from farm

The effect of 'pouring' grain from a loading bucket and movement during transit make it important to ensure effective sampling at the point of delivery. Internationally recognised standards exist for sampling lorries (see page 14), which specify numbers of separate samples required from different load areas.

If samples have been accurately taken during loading from the farm and one representative sample has been provided to the merchant/processor, this sample could, in theory, suffice for all quality assessments on delivery. However, unless a vendor assurance programme is in place, all loads delivered to store or processor will be sampled, usually using automated systems.

#### Action:

- Every effort should be made to sample grain safely at the point of outloading to provide a representative sample of the grain leaving the farm
- Check the contract, merchant annual terms or confirmation note for any special terms relating to sampling
- Sample lorries at the point of delivery according to standard protocols to produce a 2.5–4kg aggregate
- Thoroughly mix and sub-sample grain taken from lorries at intake

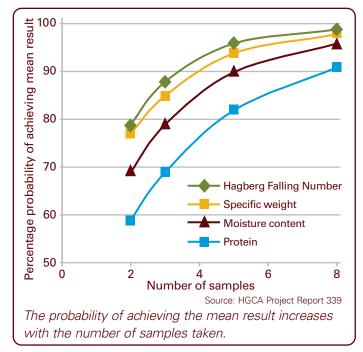


## Predicting average values by sampling

Grain within any 'lot' (trailer load, bin, floor store, lorry, etc.) can be defined in terms of moisture content, specific weight, varietal purity, protein content and many other characteristics. However, grains are not identical and grain sampling seeks to enable a representative assessment of the characteristics of the lot.

Any one sample from a single point in a grain lot would probably be unrepresentative of the characteristics of the total lot. A sampling programme aims to take sufficient samples to reduce the variability and provide a representation of the total lot. The more samples that are taken, the closer the average will be to accurately reflecting any characteristic.

Best practice is to take a series of incremental samples from different parts of any given lot, blend them thoroughly and then take a sub-sample that can be analysed to provide an average result to describe the relevant quality characteristics of the lot.



# Sampling at harvest

## Why?

Taking samples as stores are filled provides the best opportunity to generate a representative sample. Aim to create a large aggregate sample (held in a clean dustbin or similar container) formed from the incremental samples taken as the store is loaded, which can be used by grain buyers to assess likely market potential. Any sample drawn from this aggregate must be representative of it, so it is important to mix and subsample using an appropriate method (see page 17).

## Moisture content

The first priority is measuring moisture content to determine what drying is necessary by sampling loads from each trailer for moisture content. Under certain conditions of temperature and moisture content, the production of the mycotoxin, ochratoxin A, may occur. Systematic prioritisation of drying is vital.

The HGCA safe storage time calculator will help to identify grain in most urgent need of attention. www.hgca.com/grainstorage



Once the drying process is underway, further sampling helps monitor progress towards the drying targets.

## How?

Samples should be taken to represent identifiable units (lots) of grain in store. In practice, bearing in mind time constraints and modern storage practice (eg bulk storage on floor and 'pushing up' often to depths of in excess of 6m), the target is to have at least two 1kg samples that represent a unit of 100t of stored grain.

This is achieved by taking many small individual samples (incremental samples) to form an aggregate sample – a large sample comprising all smaller samples. This will be much larger than the 'final' samples retained as representative samples. The aggregate sample should be thoroughly mixed and subsampled (see page 17) to give the final representative sample.

If a bucket loader is used after grain conditioning to move grain into a bulk, representative samples can be taken using an automatic bucket sampler (see page 13).

## Safety

Reversing and tipping trailers pose a safety risk. Only sample from stationary trailers and ensure drivers are aware of your intention. It may be safer to sample tipped heaps than tipping trailers.

## Quality

To determine quality, it is best to collect samples after drying and cleaning. Samples taken as the grain goes into the store will be more representative than those taken from the bulk once the grain is in store. The only exception is for any contaminant produced during storage (eg insects or storage mycotoxins).

## Field mycotoxins

If the mycotoxin risk assessment indicates that field mycotoxins may be a problem or if the buyer has a requirement for confirmatory analysis of toxin levels, it is at this farm intake stage that representative samples should be taken. See page 16.



## Labelling the samples

Whichever method is used, key information about the samples must be recorded by effective labelling of separate retained samples. Ideally, this should include:

- Owner of the grain
- Farm name
- Store name
- Quantity represented
- Origin (field) optional
- Position in store (eg back left corner) or Bay/Bin identification
- Date sampled
- Variety
- Moisture content (in store)
- DON risk assessment score

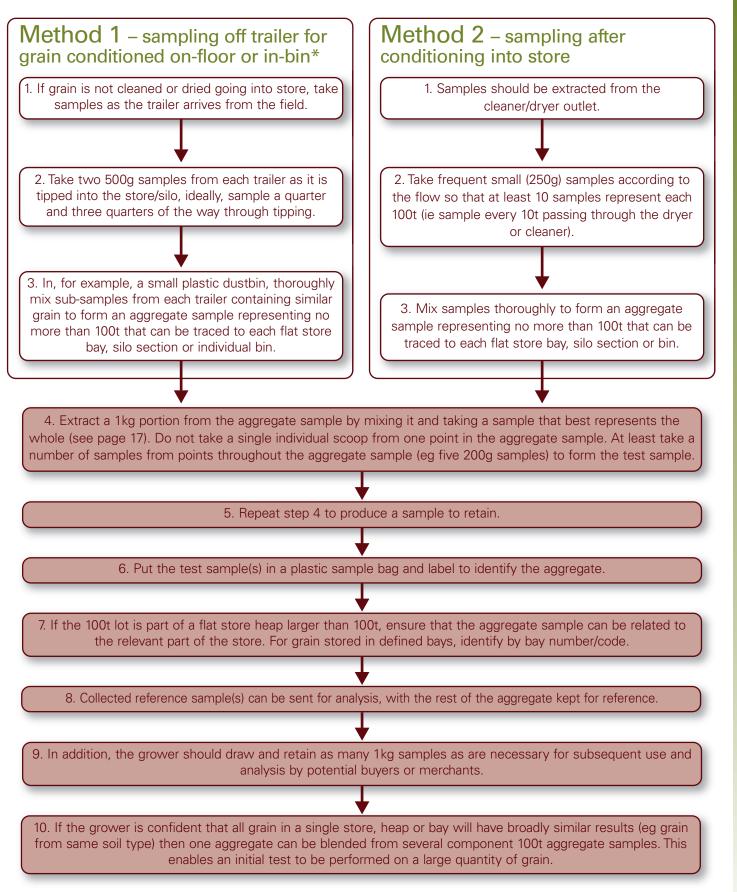
## Sample storage

Samples taken from a trailer before batch or continuous flow drying can be discarded.

Samples taken after drying, or from trailers prior to on-floor drying, can be retained for potential buyers to use to assess marketability. For longer-term storage, ensure samples are sufficiently dry and kept in rodent-proof containers or store room.

There is no need to retain samples of stored grain once all potential buyers have had the opportunity to take samples, provided each load leaving the farm is sampled.





\* If grain is wet (over 16% MC going into store), Method 1 can still be used but samples need to be dried gently before 'bagging' to avoid deterioration. Spread samples thinly on a large sheet of paper or polythene and leave overnight in a warm place. Unless grain is at VERY high moisture (when on-floor drying is unlikely) this should be adequate to dry samples for testing.

Sampling at harvest

# Sampling and monitoring

## Why?

During storage, grain is still a living 'crop' – respiring and susceptible to infection by moulds and infestation by pests. Monitoring temperature and moisture content provides early warning of any change that may threaten crop quality.

## Moisture

## Regular sampling for moisture content confirms whether drying targets are likely to be met.

High moisture can allow mites and moulds to develop. The recommended level for longer-term storage is below 14.5% moisture content for cereals and 7.5–8% for rapeseed. This is particularly important to prevent storage fungi (eg *Penicillium* species) from producing ochratoxin A.

The impact on risk of any particular temperature and moisture content combination can be assessed using the HGCA safe storage time calculator. www.hgca.com/grainstorage



## Temperature

Regular sampling for temperature confirms whether the temperature/time targets are on track and identifies any temperature rise that might indicate heating caused by insect activity or water leaks.

Most storage insect pests cannot breed below 15°C and none can breed below 10°C. However, target temperatures need to be achieved within a defined period to reduce grain temperatures below insect breeding thresholds before one life cycle is completed. As grain will come into store well above these temperature thresholds, regular monitoring of stored grain is important to follow the temperature reduction set out in the HGCA Grain storage guide.

## Pests

#### Regular sampling for pests enables early action to be taken to overcome any risks identified.

Insects (primarily storage beetle pests) and mites can cause rejection in the marketplace. They can be prevented from establishing in stored grain by conditioning grain to reduce moisture and temperature. Moisture is of primary importance for mite breeding; temperature for insect breeding.

## **Mycotoxins**

If grain has been harvested 'dry', or has been dried quickly, storage mycotoxins (such as ochratoxin A) should not be a risk, unless water has contacted the grain from the store structure.

Legally, all producers should know whether grain for human consumption meets the food safety standards for the presence of the fusarium mycotoxins, DON, ZON, T2 and HT2 (see page 16). Many processors will require this information, especially at the start of each new harvest year. Samples taken in store may be used to provide this information.

## Quality

Key quality parameters will need to be assessed by tradeassured testing facilities which may be provided by a merchant or other independent testing laboratory. Depending on the market, such tests may include:

- Protein/nitrogen quantity
- Specific weight
- Hagberg Falling Number
- Germination

Most quality parameters do not change during storage. There is, therefore, no need to extract samples for such analyses if effective sampling has taken place at intake and the location of individual sampled loads (or lots) can be accurately identified within the store.

Where prior sampling has not been undertaken, many purchasers/potential purchasers will wish to assess quality in-store using, for example, a multi-aperture spear (ideally 3–5 apertures). Such sampling is less likely to be representative of a given bulk than samples taken as the store is loaded – a spear just 1.5–2.0 metres long cannot reach through deeper bulks/bins.

## Safety

- Anyone sampling from a grain bulk should be accompanied
- Attention should be paid to the presence of loading equipment, lorries and/or trailers
- No one should enter a closed silo

For more information on grain storage, see HGCA's Grain storage guide for cereals and oilseeds www.hgca.com/grainstorage



## in store



## How?

## Moisture

When grain is dried 'in situ' (ie on-floor or in-bin), this is generally achieved by passing a high volume airflow up through the grain. This effectively 'pushes' a drying front up from lower layers towards the surface – a process that takes up to two weeks.

Moisture sampling monitors progress of the drying front. This can be achieved by sampling and testing grain near the top of the bulk/bin or with an automatic sensor inserted into the grain. The information gathered confirms when the drying front has completed its movement and drying equipment can be turned off.

Monitor for moisture content at several locations (the same each time). An increase in moisture content in a localised area of 2% or more in a week may indicate condensation, leaks, hot spots or insects. Record moisture content at least once each month during winter.

Annual calibration of moisture meters is essential. Errors are frequently +/- 0.5% and can be greater in very wet, very dry or freshly harvested grain. Take as many samples as possible and determine moisture content without delay. Keep samples in a watertight container with minimum free air space and at a steady air temperature. Mix each sample thoroughly before testing.

## Temperature

Monitor every few days until the target temperature is reached and then every week. Always record at the same location. Measurements must be taken where cooling takes longest, eg furthest from the fan in blowing systems, usually 0.5m beneath the surface and centrally between ducts.

In larger stores, consider permanent temperature probes installed in a grid pattern across the bulk. Modern installations enable constant remote monitoring, including via smartphone. Electronically recorded data also allows trends to be observed over time.

Any temperature rise over 1°C should be investigated, as it could indicate the presence of fungi, sprouting, development of pests or a leaking roof.

## Pests

While pests in stored grain can be detected by examining physical samples of grain drawn from a bulk or bin, it is a very laborious process. Traps have been shown to be more than ten times as effective as sampling at detecting low level populations of insects and mites. Some traps are designed for use in the store, others for use within the grain bulk.

Positioning traps both on the grain surface and approximately 5–10 cm below (approximately 4–5 metres apart) will provide the greatest likelihood of detecting all species of stored product insects and mites at the earliest opportunity.

Monitor for pests weekly until grain reaches the target temperature and then monthly, providing it remains at the target temperature, until spring when temperatures rise and insects become more active. Then revert to weekly monitoring. Traps should be accounted for each time they are examined and a permanent record of the contents should be kept.

It is important that any pests found are correctly identified. Refer to the HGCA Grain storage guide or the poster: Insects and mites in stored grain and grain stores. www.hgca.com/grainstorage



## Record-keeping

Keeping a record of each monitoring activity both provides evidence of due diligence and allows trends, particularly in temperature and moisture, to be spotted.

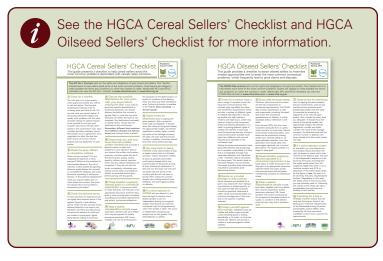
Electronic systems provide a simple way to record all measurements and assessments made during storage. As an alternative to time-consuming probing or sampling grain, fixed probes/sensors can be installed in stores to monitor conditions in store constantly (eg moisture and temperature).

# Sampling at outloading

## Why?

Best practice, to which the industry should aspire, is to take and retain samples from each lorry load before it leaves the farm. These samples will not be contractual for the purposes of the determination of quality or condition at the delivery point but they may help if problems arise.

The quality and quantity of grain leaving the farm represent the result of a year's work and this is the best opportunity for the grower's assessment and the purchaser's assessment of delivered quality to match. Taking and retaining samples is also a requirement of some assurance schemes.



## How?

The sampling method depends on the equipment used for loading the lorry. In all cases, sampling should follow standard protocols to maximise the validity of retained outloading samples.

For a 30 tonne lorry-load of grain, take at least 10 samples of 200g as the grain is loaded. This provides a 2kg aggregate sample.

## Safety

- Do not attempt to climb on top of lorries, unless there is special provision
- Be aware of risks around loading equipment and vehicles at outloading
- Wear hi-vis clothing at all times when working around loading lorries

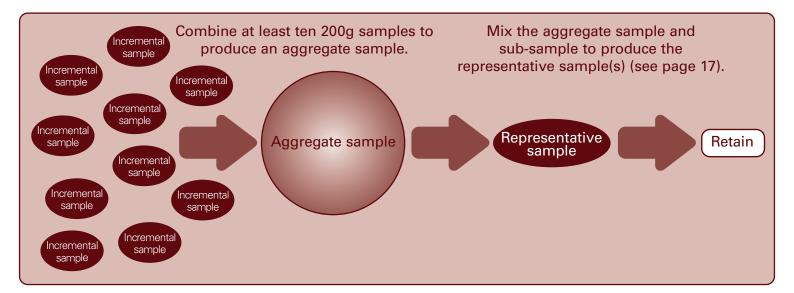
## Labelling the samples

- All samples must be labelled with basic information, including:
- Farm name
- Store name/number
- Bin number
- Variety
- Date
- Time
- Vehicle registration and trailer number

## Sample storage

Samples should be stored in airtight containers (for example, polythene bags or plastic boxes) in a cool, dry place safe from rodent attack.

These samples should be retained until payment has been received for the loads to which the samples relate.





## Sampling from the loading bucket

Sampling from the loading bucket is the best method to obtain a representative sample but manual sampling ideally requires a second person. A safer method would be to use an automatic bucket sampler.

- Scoop samples from the grain in each bucket loaded
- Combine these incremental samples into a single 2kg aggregate sample in a bucket
- Mix the aggregate sample and divide into two 1kg representative samples (see page 17)



## Sampling from spout loading

For grain being loaded into a lorry from a conveying system, it is best that grain is sampled at a point close to the loading location, where samples may be drawn safely. In some cases, an 'interrupter' plate can be inserted into conveying tubes.

- Collect at least ten 200g samples
- Combine these incremental samples into a single 2kg aggregate sample in a bucket
- Mix the aggregate sample and divide into two 1kg representative samples (see page 17)

#### Sampling from a grain heap

When loading bulk/on-floor stored grain, the grain needed for the next lorry could be pre-positioned into a separate heap. This can be sampled with a conventional grain spear preferably multi-aperture).

- Take at least ten 200g samples
- Combine these incremental samples into a single 2kg aggregate sample in a bucket
- Mix the aggregate sample and divide into two 1kg representative samples (see page 17)



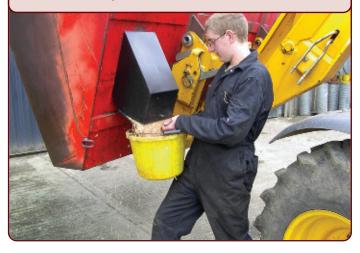
Automatic bucket sampler

Manually collecting a sample from each bucket as it is loaded is time-consuming. Automatic samplers are available that extract a small amount of grain from each bucketful during the process of loading a lorry.

Such samplers collect a maximum of between around 0.8kg and 2kg of grain until full. It is important to choose a product that can ultimately deliver two 1kg subsamples for each lorry-load. If less than 2kg is collected, while still a representative sample, it is likely to give less accurate results than a larger sample.

The sampler is robust and simple with no moving parts. All the individual bucket samples collected as the lorry is loaded are mixed in the sampler to form an aggregate sample. All the operator has to do is empty the sampler after each lorry has been loaded and bag the samples.

In HGCA-funded tests, this sampling technique proved easy to use, did not delay loading and did not compromise operator safety. The test results of 'bucket' samples agreed well with samples collected from lorries at delivery when best practice recommendations were followed by both the farmer and the receiver.



# Sampling at commercial i

## Why?

All processors of any size sample and analyse grain at intake to determine its quality and whether it has met the agreed contractual requirement and specifications. Additionally, most will test at intake for the presence of mycotoxins, particularly at the start of the new harvest season.

The testing done by processors may be quantitative (eg moisture, protein and Hagberg Falling Number) or qualitative (eg taint or visual appearance). Different delivery points (animal feed manufacturers, millers, maltsters, breakfast cereal manufacturers, exporters, etc.) will carry out different tests to assess quality but sampling methods will tend to be similar, especially in larger premises.

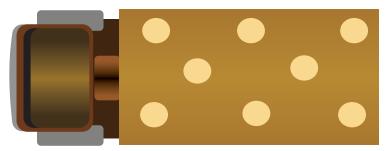
The biggest sampling issue with incoming loads of grain is that quality across the vehicle may not be uniform. Vehicles are sometimes loaded with grain taken from different locations in the grain store and the stored grain may not be uniformly mixed. Heaping in the vehicle, caused by bucket loading, does not always level out during haulage and this can bias sampling.

## How?

The AIC No. 1 contract states that deliveries shall be sampled by the receiver at the final consignment point in accordance with the procedure laid down in the ISO 24333 Standard or any any amendments to it. Check the contract, merchant annual terms or confirmation note for any special terms relating to sampling.

The example below is the recommended ISO sampling method for a 30 tonne bulk vehicle load of grain at an intake point (ISO 24333:2009). The circles represent where a spear sample should be taken.

- Ideally, eight samples should be taken (three samples for lorries of 15 tonnes or less)
- The spear must be long enough to sample the whole depth of grain
- Sampling position is more important than the number of samples taken from each load: the lorry should be positioned so that most of the load is accessible



## Automatic samplers

Virtually all medium to large processors sample grain on arrival using a mechanised spear that is programmable or is controlled from the laboratory or intake office via a joystick. The spear draws a sample up through the core of the load, having sensed the bottom of the trailer. This minimises the amount of fine/small grains in the sample, which would adversely bias the amount of screenings in the sample.

In flight from the lorry to the laboratory, separation of the grain is likely to occur; the smaller grains and screenings will arrive earlier than the larger grains. It is, therefore, particularly important to mix the sample thoroughly before taking analytical samples for testing.



#### Hand-held samplers

Smaller processors may sample using handheld manual spears. These may also be used at larger facilities when breakdown of the automatic sampler occurs.

If sampling manually, a spear should be used that will collect samples from different depths and collect about 500g per sampling point – this may require more than one insertion per sampling point.



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#### Mixing and sub-sampling

Most automatic sampling equipment produces an aggregate sample of 6–8 kg. To ensure representative subsamples are used for testing, this sample must be thoroughly mixed, using methods similar to those described on page 17.

#### Cleaning

Companies will have a policy on whether screenings and admixture should be included when a sample is tested. This should be made known to the seller and form part of the contract. This will take into account whether the calibrations for specific tests were developed using clean samples and whether admixture could cause damage to or block equipment.

Cleaning can be carried out using sieves, for example, those used to determine screenings, or more specialist equipment may be used. Samples should be labelled to indicate whether or not they have been cleaned.

#### Grinding

For some methods, whole grains are not suitable and the sample is ground. Individual grains with extreme values will affect the result if small samples are used. To reduce this effect, a relatively large sample should be ground.

Most grinders cause particle separation. Bran or husk grind at different rates to endosperm and are often on the surface of the ground sample. Mixing of the ground sample is essential.

To prevent cross-contamination, grinders should be cleaned or flushed between samples and cleaned thoroughly at the end of the day.

For information on the visual inspection of grain, see the HGCA poster 'Inspecting grain for defects and impurities'.

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#### Storage and labelling

Sample containers must limit moisture loss and avoid cross-contamination with other samples. For storage, containers should be full and plastic bags should have the air expelled from them to prevent moisture loss and spillage.

Adequate labelling is important to allow samples to be traced and the results assigned to the correct samples. Labelling of lids should be avoided, as these can be swapped between samples. The length of time samples are retained will depend on company policy and/or contracts.



## Laboratory testing

Exact testing requirements will be specified in contracts but, for some tests, a reference procedure may be used (eg ISO 712:2009 for moisture determination). Most flour mills and maltings will use modern alternative equipment for some tests (such as NIR) but these will be traceable to the reference procedures.

Flour mill intake laboratories take part in the **nabim** proficiency scheme (or equivalent), maltings participate in the Malting Analytes Proficiency Scheme (MAPS) and TASCC laboratories also take part in proficiency schemes to ensure conformity of results throughout the year.



See www.hgca.com/graintesting for more information and training tools for grain analysts.

# Sampling and mycotoxins

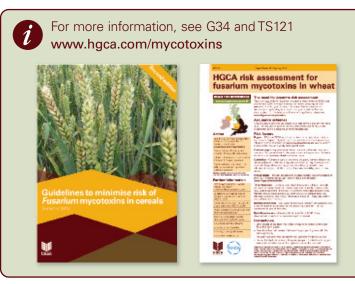
## **Mycotoxins**

Mycotoxins are toxins produced by fungi that infect crops and/or grow on grain.

In cereals, contamination by mycotoxins can result from either field-borne infections or from fungi that develop in stored crops.

## Field mycotoxins

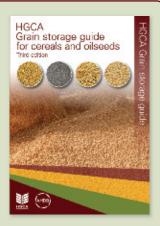
- Infection of ears by *Fusarium* species can result in mycotoxin development
- The HGCA risk assessment for fusarium mycotoxins in wheat should be completed
- Mycotoxins formed before harvest are stable and likely to remain during storage but not increase



## Storage mycotoxins

The main fungus with the potential to cause problems in stored grain in the UK is *Penicillium verrucosum*. Under certain conditions (18% moisture content and above), this fungus can produce the mycotoxin ochratoxin A.

For more information on grain storage, see HGCA's Grain storage guide for cereals and oilseeds www.hgca.com/grainstorage



## Legal limits

Legal limits exist for the three principal mycotoxins (DON, ZON and ochratoxin A) that may occur in cereals and cereal products intended for human consumption. Guideline limits also exist for cereal intended for animal feed.

The EU Commission has now published a Recommendation for T2 and HT2 establishing indicative levels. It also sets out requirements for industry and authorities in terms of monitoring against these levels and what investigations must be undertaken when repetitive results above the indicative levels are found. These levels are not, however, maximum limits, nor is there any suggestion that they represent a risk to food safety.

It is the responsibility of all food business operators (including farmers, merchants and processors) not to place on the market any cereal or cereal products that exceed the legal limits. This means that all sellers must be able to demonstrate due diligence in determining the levels of mycotoxins that are present.

The current legal limits for the principal mycotoxins in grain for food markets are:

- Deoxynivalenol (DON): 1,250 ppb (unprocessed wheat and barley), 1,750 ppb (unprocessed oats)
- Zearalenone (ZON): 100 ppb (unprocessed wheat, barley and oats)
- Ochratoxin A (OTA, OA): 5 ppb (unprocessed cereals)

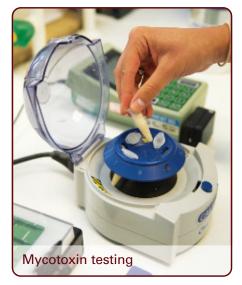
Individual processors may set contractual maximum levels, depending on the type of product, such as baby foods or breakfast cereals.

## Sampling for mycotoxins

Sampling specifically for mycotoxins requires a demanding regime because the distribution is not likely to be uniform within a stored bulk. Fungal growth and associated mycotoxins

can be very localised, especially for the storage mycotoxin, ochratoxin A.

The sampling protocols in this guide have been designed to meet the more demanding requirements of sampling for mycotoxins, as well as other aspects of grain assessment and monitoring.

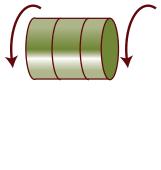


# Mixing and sub-sampling

A large aggregate sample of grain will include grains of different sizes and quality. It is important to ensure, as far as possible, that all grains in the aggregate have an equal chance of being included in any sub-sample drawn from it.

#### Step 1. Mix the large aggregate sample as thoroughly as possible.

This can be achieved by using either a large drum with a securely fitting lid or by hand-mixing the aggregate on a clean floor area.



If using a **drum mixer**, ensure it is no more than half full, roll the drum along its axis and invert it at least five times. Sub-samples of around 1kg can then be extracted by taking, for example, five samples of approximately 200g from different positions in the drum.

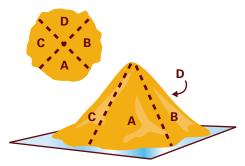


If mixing by hand, after tipping the grain from its original container holding the aggregate (eg a large plastic dustbin) onto the floor, it can be mixed using a clean shovel/scoop.

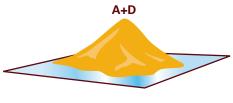
## Step 2. Produce the representative samples.

A number of methods of varying complexity can be used for sub-sampling an aggregate sample after it has been thoroughly mixed. These include 'coning and quartering' and sample dividers, such as a cone-shaped divider, a rotary mechanical divider or a riffle divider. Details for using such equipment are given in ISO24333; the only constraint is that riffle dividers should not be used for samples of less than 2kg. The 'coning and quartering' method is described below.

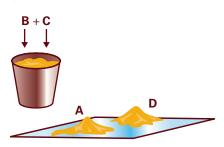
#### **1.** The heap of tipped grain will take the rough form of a cone – it can be described as having four quarters.



**3.** Mix samples A and D again to form a new cone of grain.



2. Select two opposite corners (eg A and D) and remove the other two quarters and return them to the original container.



**4.** Repeat until the size of one of the quarters is the equivalent weight of the final sample required (eg 1kg).





Note: when retaining selected quarters, it is imperative that ALL the material in the quarter, including admixture, is collected.

# Further information

## **HGCA** information

HGCA publications and details of HGCA-funded projects are all available on the HGCA website – www.hgca.com

## Grain storage

- **G52** HGCA Grain storage guide for cereals and oilseeds, 3<sup>rd</sup> edition (2011)
- **G56** Rodent control in agriculture an HGCA guide, 2<sup>nd</sup> edition (2012)
- G37 Grain moisture guidelines for measurement (2008)
- P01 Insects and mites in stored grain and grain stores (poster)

## Grain testing

- P06 Inspecting grain for defects and impurities (poster)
- G21 Grain testing standards for testing (2004)

## **Fusarium mycotoxins**

- **G34** Guidelines to minimise risk of fusarium mycotoxins in cereals (2010)
- **TS121** HGCA risk assessment for fusarium mycotoxins in wheat (2013)

## Sellers' Checklists

- CSC2012 HGCA Cereal Sellers' Checklist (2012)
- OSC2013 HGCA Oilseed Sellers' Checklist (2013)

## **HGCA Project Reports**

- PR515 Investigation into the specific weight differences between wheat varieties tested in the HGCA Recommended List and commercially-grown crops from 2007–2012 (2013)
- **PR514** Survey of current agronomic practices influencing free fatty acid content in oilseed rape during the 2011/12 season (2013)
- **PR510** Ensuring that UK cereals used in malting, milling and animal feed achieve food and feed safety standards (2013)
- PR464 Food safety review of UK cereal grain for use in malting, milling and animal feed (2009)
- PR426 Research to develop practical user guidelines to maximise the accuracy of moisture meters (2008)
- PR407 Assessment of three commercial automatic grain samplers fitted to front loader buckets (2006)
- **PR362** Development and validation of on-farm sampling protocols: Assessment of an automatic bucket sampler for use during out-loading (2005)
- **PR349** A national grain sampling and analysis system for improved food marketing and safety (2004)
- **PR339** Grain sampling and assessment: Sampling grain in lorries (2004)
- **PR325** Developing and validating on-farm sampling protocols: sampling in store and during outloading (2004)
- **PR301** Development and validation of on-farm sampling protocols for collection of marketing (quality) samples at harvest (2003)

## **HGCA** Research Reviews

**RR50** Grain sampling methods to achieve consumer confidence and food safety (2003)



nabim Publication

Wheat and flour testing (nabim, 2011)

## **Rural Payments Agency Publication**

Explaining the Common Agricultural Policy of the European Communities for the Basic Methods for Sampling Cereals (Leaflet Number IM(C)18, August 2013) www.rpa.defra.gov.uk

## Websites

Agricultural Industries Confederation (AIC): www.agindustries.org.uk

BSI Standards: http://shop.bsigroup.com

Federation of Oils, Seeds and Fats Associations (FOSFA): www.fosfa.org

Grain and Feed Trade Association (GAFTA): www.gafta.com

HGCA: www.hgca.com

Maltsters' Association of Great Britain (MAGB): www.ukmalt.com

National Association of British and Irish Millers (nabim): www.nabim.org.uk

NFU: www.nfuonline.com

Red Tractor Farm Assurance:

www.assurance.redtractor.org.uk

Scottish Quality Cereals (SQC): www.sfqc.co.uk

## British and International Standards BS EN ISO 24333:2009 Cereals and cereal products – sampling

This International Standard is often quoted by the UK's grain industry quality schemes and by trade sampling standards.

In its introduction, the Standard describes sampling as a process needing methods and equipment suited to the purpose. Quite rightly, as any interpretation will be futile if the sample failed to represent the lot appropriately.

This is why sampling is so important and should only be entrusted to correctly trained people with equipment that is suited to the task.

The Standard runs to over 28 pages and the following summary of points is suggested as relevant to the UK grain trade.

- For static bulks, manual probes can only be used to a depth of 2m. For grain stored deeper than 2m, only mechanical or suction sampling devices should be used.
- Also for static bulks (to include silos or warehouses) a minimum of 15 individual samples must be taken to provide a laboratory sample to represent 100t of a static grain bulk. For smaller lots, eg lorries up to 30t, eight samples must be taken and each individual sample must weigh a minimum of 400g.
- 3. Sampling grain for contaminants requires a different sampling frequency/total aggregate size than for other analyses. As examples, 1–3kg is the minimum mass for the laboratory sample used for other analyses; 10kg is required for ochratoxin A; 1kg for pesticides, heavy metals, dioxins, etc.; and 3kg for other contaminants (except DON/ZON, etc. that can be of 1kg).
- 4. No laboratory sample should weigh less than 1kg.
- 5. All laboratory samples must be thoroughly homogenised.

#### BS EN ISO 542:1990 Oilseeds – sampling

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. Stoneleigh Park Warwickshire CV8 2TL www.hgca.com

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Photographs of automatic bucket sampler (page 13) courtesy of Claydon Yield-O-Meter Ltd.

Edited by Dr Emily Boys, HGCA.

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