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Cereals and cereal products — Sampling studies

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European foreword

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Full document details: PD CEN ISO/TR 29263:2021

Contents

Page

Foreword ... **iv**

Introduction ... **v**

1 Scope ... **1**

2 Normative references ... **1**

3 Terms and definitions ... **1**

4 Context ... **1**

5 Study n°1: extract from "Grain sampling and assessment: sampling grain in lorries" – Project report n° 339" ... **1**
- 5.1 General ... 1
- 5.2 Context ... 1
- 5.3 Studies conducted and objectives ... 3
- 5.4 Methodology ... 3
  - 5.4.1 Conducting tests ... 3
  - 5.4.2 Results and conclusions ... 5

6 Study n°2: extract from "Sampling grain in static and flowing condition – alternatives to the regulatory protocol" ... **19**
- 6.1 General ... 19
- 6.2 Context ... 20
  - 6.2.1 Regulatory aspect ... 20
  - 6.2.2 Normative aspect ... 20
- 6.3 Studies conducted and objectives ... 20
  - 6.3.1 Study A ... 20
  - 6.3.2 Study B ... 21
- 6.4 Study A: silos and lorries of wheat and corn – Fusariotoxins and quality assessment ... 21
  - 6.4.1 Organising field tests ... 21
  - 6.4.2 Results and conclusions ... 33
- 6.5 Study B : silos of corn – Fusariotoxins; flowing grains ... 48
  - 6.5.1 Organising field tests ... 48
  - 6.5.2 Results and conclusions ... 49

7 Study n°3: extract from "Investigation of the distribution of deoxynivalenol and ochratoxin a contamination within a 26-T truckload of wheat kernels" – Project report ... **90**
- 7.1 Context ... 91
- 7.2 Methodology ... 91
  - 7.2.1 Instruments for sampling and sample homogenization process ... 91
  - 7.2.2 Reagents and materials ... 91
  - 7.2.3 Sampling procedure ... 92
  - 7.2.4 Sample comminution ... 93
- 7.3 Results and Discussion ... 93
  - 7.3.1 Distribution of DON and OTA infected kernels within the lot ... 93
  - 7.3.2 Sample comminution study for the dry milling process ... 95
- 7.4 Uncertainty comparison on the basis of number of incremental samples ... 95
- 7.5 Conclusions ... 97

Bibliography ... **98**

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Full document details: PD CEN ISO/TR 29263:2021

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Introduction

This document presents the results of three groups of studies which results have been used to elaborate ISO 24333

These studies have been managed by United Kingdom in May 2003, by France in 2004-2005 for the first one and 2006-2007 for the second one and Germany in 2008.

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Cereals and cereal products — Sampling studies

1 Scope

This document presents the description and the results of the three studies conducted by United Kingdom, France and Germany related to grain sampling in order to define a harmonized sampling protocol for official controls.

These results had been used to draft ISO 24333.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Context

European directives for official controls of some contaminants such as mycotoxins required methods for sampling and analysis. In order to harmonize sampling procedures necessary for these analysis and to determine the best way to prepare a homogenous and representative laboratory sample, studies had been conducted by United Kingdom, France and Germany.

The results of these 3 studies are presented in this report.

5 Study n°1: extract from "Grain sampling and assessment: sampling grain in lorries" – Project report n° 339”

5.1 General

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This is the final report of a 13-month project that started in May 2003. The work was funded by HGCA (project 2955).

5.2 Context

This two-year programme was made at the request of HGCA to improve and standardise grain sampling and analysis across the UK cereals industry.

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The first phase of the programme was to develop and validate protocols suitable for collecting samples of grain on UK farms at harvest time and to train farmers in their use. The second part was to examine approaches to the collection of samples during storage and to compare the results obtained, wherever possible, with data collected as the store was filled. During the course of earlier sampling work, there was strong interest expressed in the mechanics and effectiveness of sampling loads of grain in lorries. In addition, some of the work done during storage involved sampling grain as it left the store in lorries. This showed up the limitations of some approaches to lorry sampling and highlighted the need for more information. An assessment of lorry sampling was therefore, added and is reported here.

Almost all grain is sampled as it is delivered to end users to confirm its quality and to ensure that contractual obligations are met. This sampling takes the form of collecting one or more samples from a lorry-load on arrival. The equipment used and method of sampling varies between end users and there are no data to show if these variations may cause bias in the representativeness of the sample and, therefore, in the results of quality analysis.

The key aim of end-user sampling is to ensure that the quality of the grain is suitable for its intended use. Therefore, sampling is done before the load is tipped and this limits access to the surface of the load. This access is further constrained by food safety and HSE legislation that prevent the sampler from walking on the load.

A limited assessment of the practicalities of sampling lorries was done in 1992 (HGCA Project Report 79) in which the effects of method of sampling, number and position of sample points, the methods of loading lorries were considered. The wheat sampled was low-grade feed material with a low specific weight and a high level of fine material so was not representative of other grades. The results suggested that loading lorries with a front loader or from a hopper had no effect on the distribution of the quality characteristics within a load. Small differences were noted in the mean values for specific weight between automatic sampling using a Samplex CS90 and a manual spear but overall variability of the grain meant that these differences were not significant. There was significant variability in the results obtained at individual points with either method, although this variability was random and not associated with any part of the load. Fine material appeared to be very difficult to measure. At the time, this work was undertaken there were no restrictions limiting access to the surface of the grain so that a widely disbursed pattern of sampling points could be used with the manual sampling. The conclusions from this work were that it was extremely unwise to base an assessment of lorry load of grain on a single sample and that more work was needed to confirm the results and to assesses other grades of grain. The aim of this project was to establish if there are any inherent problems with the sampling of grain for the determination of quality characteristics in lorries at the point of intake and to establish recommendations in the form of a protocol for the sampling of grain under these conditions.

Grain was sampled using automated systems (Samplex CS90) and manual spearing to see if the method of sampling influenced the grain quality measurements. A key part of the process was to assess the influence that the number of samples taken from each load had on the likely accuracy of the results. Samples were collected at 4 different locations; on two occasions 10 lorries were sampled and on two occasions 8 lorries were sampled. At three locations, CS90 samplers were used and 8 samples were withdrawn from each load and at the other location samples were taken manually with a multi-compartmented spear with 5 samples being taken from each lorry. A comparison of different ways of sample handling was obtained by comparing the individual results from the 8 samples against an analysis of samples withdrawn from a composite sample formed from 8 samples. The latter method reflects more accurately the procedure followed at most stores.

Results indicated that there were no statistically significant differences between results from the individual samples or from the composite samples. Monte Carlo simulation of the impacts of using 2, 3, 5 or 8 samples per load revealed that the greater the number of samples used the greater the reliability of the result and the more likely it was to represent the true mean of the load. It was noted that automatic sampling equipment can no longer sample the entire length of a trailer and this could cause problems with obtaining the ideal sample. Manual sampling also had severe limitations due to the lack of safe access for sampling of trailers.

A sampling protocol for lorries is presented which emphasises the need for 8 samples to be taken from each load in order to get a good representation of the quality of the entire load.

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5.3 Studies conducted and objectives

The study was conducted to assess the effectiveness of different approaches to sampling loads of grain in lorries.

The specific objectives were:

— To assess if the method of collecting samples influences grain quality measurement;

— To assess if the number and position of sampling points influences grain quality measurement;

— To provide guidelines for sampling lorries giving reliable information about grain quality.

5.4 Methodology

5.4.1 Conducting tests

5.4.1.1 Collection of data relating to current sampling practice

In November 2002 the HGCA circulated a questionnaire to commercial grain stores and end-users of grain requesting information about their methods of analysis and methods of intake sampling. The information that was collected was used to assist with the design of the assessment of lorry sampling.

5.4.1.2 Sample collection

5.4.1.2.1 Store 1

The work was done at a store specialising in the storage of malting barley. Lorries were loaded with malting barley, variety Pearl, of a quality representative of that delivered to central storage from farms. The lorries, all 28 t articulated units, were loaded with a front loader fitted with a 2 t bucket.

Ten loads were sampled over a 2-day period. Sampling was done using the store's Samplex CS90 automated vacuum sampler. Initially, it had been expected to re-programme the CS90 to take 10 samples/load in a pre-set pattern. However, observation of the method of operation and sampling pattern achieved by the CS90 suggested that there was no advantage in using more than the 8 points provided by one of the standard sampler programmes.

During the setting up and initial testing of the CS90, the slide on the sample spear was opened to its maximum to increase the sample size. The system was set to collect grain only during the withdrawal as is recommended by the manufacturer for granular materials.

Each of the eight points was sampled three times. On the first occasion, individual samples were held separately. During the second and third samplings, all samples were bulked into single batches. One of these bulk samples was held as a composite sample and the other was used to provide samples of 1, 2 and 3 litres (small medium and large) collected at random with a 1-litre jug.

A small sub-sample from each of the individual samples was tested on the spot by store staff for screenings and germinative capacity. Screenings were tested by sieving a 100 g sub-sample with a motorised shaker fitted with a 2,25 mm mesh screen for 2 minutes. The germinative capacity was tested using the standard tetrazolium test.

5.4.1.2.2 Store 2

Work was done at a commercial store during the normal out-loading of feed wheat. The lorries were loaded from an on-floor bulk using a front-bucket loader and were sampled as they left the store. Normal sampling practice was to collect a single spear sample/load using a manual, multi-compartmented spear of about 1,7 m in length. Access to the load was via a small sampling platform that only allowed samples to be taken from less than half the length of the loaded trailer and from only one side of the load.

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For the purposes of this investigation, 5 sample points were used for each load and the lorry was moved forward during the sampling process so that access to the whole length of the load could be obtained. However, it was not practical to turn the lorry round to give access to both sides of the load. Manual sampling meant that there was, inevitably, some variation in the exact location of the sample points between loads. Three spear samples were collected at each point. The first was held as an individual sample, the second bulked to form a composite sample and the third bulked to give a sample from which three random samples (small, medium and large) could be taken without mixing.

Ten lorry loads were sampled over a two-day period.

5.4.1.2.3 Store 3

The assessments were made at a commercial store during the normal out-loading of milling wheat. Samples were collected using a Samplex CS90 but without the automatic option. Therefore, the spear had to be controlled manually by the operator and this meant that there was considerable variation in the positioning of sample points between loads. A further constraint on sampling was that the CS90 was positioned at one end of the weighbridge thus limiting access to just one-half of the load. The slide on the sampler was fully open and grain was collected only as the spear withdrew.

The lorries were loaded using a bucket loader from a 2 000 t batch of wheat stored on-floor. Only a limited number of loads were dispatched each day and time constraints meant that only 8 loads were sampled during this assessment.

Three samples were collected from 8 sample points in each load. The first was held as an individual sample, the second bulked to form a composite sample and the third bulked to give a sample from which three random samples (small, medium and large) could be taken without mixing. The 8 points were arranged in a 3, 2, 3 pattern with the two samples being taken from the centre line and each row of 3 taken down the sides of the load (see Figure 1). The location of the sampler and the position of the lorry on the weighbridge meant that the samples always came from the front half of the load.

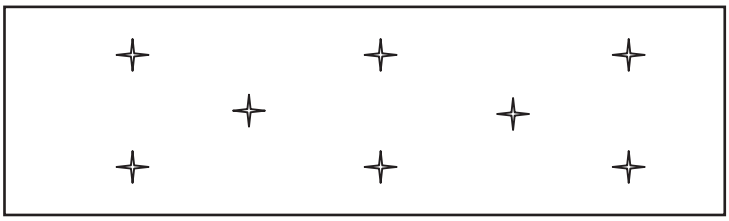

Figure 1 — Arrangement of sampling points used by CS90 sampler

Data was supplied by the storekeeper giving the store's own assessment of the quality of the batch of grain as measured when the store was filled.

5.4.1.2.4 Store 4

Loads of milling wheat delivered to a large flour mill were sampled with a Samplex automatic CS90. This work was done some months after the earlier assessments and it was decided that the collection of extra samples to make up composite batches was not justified. Eight samples were collected from eight loads of wheat over a 10 d period. For technical reasons, the collection of samples had to be done by mill staff so the exact details of the points sampled are not known.

5.4.1.3 Assessment of samples

Samples had to be transported to the testing laboratory and some additional delays occurred between the collection and assessment of samples. However, samples that could not be analysed within 48 h were stored in a freezer at -16 °C to minimise changes in the properties of the grain. These samples were allowed to return to ambient temperature before testing. The exceptions to this were samples collected from store 4.

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These were collected over a 10 d period and a further 4 d elapsed before the samples arrived for testing. During this period, the samples were not held under controlled temperature conditions.

Individual samples were tested separately, as were the three different sizes of random sample. However, in the case of the medium and large samples, these were divided by coning and quartering to give the correct volume of grain for assessment. The composite samples were also mixed and then divided by coning and quartering. Five sub-samples of the composites were tested from Store1 but only three sub-samples were tested from stores 2 and 3.

As a first step in the assessment process, the screenings in each sample were measured by manual sieving. Each sample was weighed and then sieved for 30 s using a 2,5 mm slotted sieve for wheat or a 2,25 mm slotted sieve for barley. The sievings were weighed and the percentage calculated. The weights of the individual samples gave an indication of the variation in the size of sample collected on each occasion.

After sieving, the properties of each sample were assessed using a Foss Infratec Grain Analyser 1241 GA-TWM[1)]. The machine used official calibrations as provided by the NIR network and measured moisture content, specific mass, protein in the case of wheat, or nitrogen in the case of barley and made an assessment of hardness of wheat.

In addition, some of the samples of wheat from stores 2 and 3 were sent to NIAB for assessment of Hagberg Falling Number. Complete sets of individual samples from 5 loads, together with a single composite sample were tested from Store 3. Complete sets of individual samples and a single composite from 4 loads were tested from Store 2. The testing was done using standard methodology and each result was the mean of two determinations.

5.4.1.4 Estimating the reliability of sampling

The impact of the number of samples taken on the reliability of the result obtained from those samples was assessed by determining the mean and standard deviation for the results of the 8 samples taken from each lorry. This information was used to define a probability curve for a normal distribution for each of the lorries. A Monte Carlo simulation was then run to sample from either 2, 3, 5 or 8 of these distributions depending on the sampling regime to be simulated. The simulation was run for a total of 100 000 trials. From this result, cumulative probability distribution curves were obtained and these were used to estimate the confidence limits for different numbers of samples for a given margin of error. Thus, the confidence interval for sampling 2, 3, 5, or 8 times for a known deviation from the mean for the different quality parameters could be produced. The final figure shows the probability of the confidence interval actually covering the mean value.

5.4.2 Results and conclusions

5.4.2.1 Sample analysis results

5.4.2.1.1 Collection of data relating to current sampling practice

As part of the initiative to standardise grain testing a survey of laboratory practice was undertaken and this included a set of questions on the collection of samples from lorry-loads of grain. This provided information from a range of commercial premises receiving, handling or processing grain about current lorry sampling practices. The response showed that there was no common industry-wide approach. The most frequently used equipment was the Samplex CS90 or other unspecified Samplex units (49 %) followed by manual sampling (37 %). The number of samples collected per load ranged between 1 and 10 and the mass of grain collected varied between 0,4 kg and 11 kg.

1) Foss Infratec Grain Analyser 1241 GA-TWM is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of thisproduct.

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5.4.2.1.2 Observations on the sampling

5.4.2.1.2.1 Store 1

During the assessment, it became apparent that the reach of the CS90 sampling arm and the size of current articulated trailers resulted in parts of the load being inaccessible unless the lorry was moved. Of the total 11 m length of the trailer, up to 2,5 m at the front and 2,5 m at the rear of the trailer were not accessible to the sampler. Therefore, up to 40 % of the grain could not be sampled (see Figure 2).

The weight of individual samples collected varied considerably. The size of sample was related to the depth of grain at the point of sampling, the greater the depth, the larger the sample. The bucket loading method led to peaks and troughs in the loaded grain and this, in turn produced variation in sample size (see Figure 2 and Table 1).

Each individual sample was sucked from the sample spear to the laboratory and was collected in a cyclone. There was obvious separation of fine material and grains during this process (see Figure 3).

Figure 2 — Collection of samples from lorry-loads of barley with a Samplex CS90

Figure 3 — Individual samples collected in the cyclone showing the separation of fine material

5.4.2.1.2.2 Store 2

The collection of samples was limited to one side of the load only and the exact location of the sample points varied between loads because the position of the lorries in relation to the sampling platform varied. The depth of the grain in the trailers varied as at Store 1. However, the grain was always deep enough to permit the manual spear to be inserted to its full depth so there was much less variation in the weight of sample (see Table 1). Conversely, the spear was usually not long enough to reach to the bottom of the load. The process of collecting 5 samples/load was time consuming and physically

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demanding. Moving the lorry to allow access to the whole length of the load could not be considered as a practical approach in most situations.

5.4.2.1.2.3 Store 3

The manual control of the insertion of the sample probe was imprecise because the probe tended to swing back and forth as it was moved between sample points. The swing was exaggerated by the effects of wind. The position of CS90 meant that only the front half of each load could be sampled.

The size of individual samples was related to depth of grain in the trailer and this varied within and between loads.

5.4.2.1.2.4 Store 4

The Samplex CS90 was situated some distance from the laboratory and, during normal sampling in which 8 samples were collected/load, the samples were accumulated at the CS90 and then conveyed by suction to the laboratory. The bulked samples were then passed through a mixer/divider to produce a working sample for analysis. Therefore, during normal sampling procedures, it was not possible to obtain samples from individual positions within a load. For the purposes of this investigation, 8 loads were sampled so that each of the eight individual samples/load was conveyed to the laboratory and collected before the next sample was taken. This was a time consuming and disruptive process so that it was only possible to sample a single load/day.

5.4.2.1.3 Mass of samples collected

The masses of the individual samples collected from the loads at each store are summarised in Table 1.

The wide variation in the weights of samples both between and within loads found at the two stores where grain was sampled with a CS90 relates to the variation in the depth of grain at different parts of the load. The variation did not occur with the manual spear because, irrespective of the depth of grain, the spear could always be inserted to its full length.

5.4.2.2 Analysis of the quality parameters of the grain

The results of the quality analysis done using the Foss Infratec Grain Analyser are given in Tables 2, 3, 4 and 5. The results of assessing the level of fine material (screenings) are also given. For stores 2 and 3 the amount of fine material was established by the project staff using a manual sieve but data for the additional tests done by store staff at Store 1 using a mechanical shaker are also included.

The storekeeper at Store 3 provided the project with the results of intake sampling of the store used for filling the loads sampled as part of this project. Obviously, the grain sampled during the project (~250 t) represented a very small part of the 4 500 t contained in the store. However, values are given in Table 5 that allow some comparisons to be made with the results obtained by sampling loads leaving the store after about 10 months storage (see Table 4). The intake sampling procedure used at the Store 3 was to collect samples from three points from each load and to mix these samples to give a composite that was used for analysis. The analyses were done using a Foss Infratec that was calibrated to the same standards as applied to the instrument used during the project. A summary of the storekeeper's analysis results is given in Table 6.

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Table 1 — Maximum, minimum and mean masses of the batches of individual samples collected at each store. There were 8 individual samples at stores 1 3 and 4, and 5 individual samples at store 2

		Mass g		
Store	**Load**	**Max.**	**Min.**	**Mean**
Barley Sampled with automatic CS90	1	738,7	476,8	604,2
	2	725,4	615,2	670,6
	3	808,5	469,1	654,5
	4	826,4	425,0	649,4
	5	761,9	437,4	604,9
	6	721,6	487,3	610,5
	7	816,1	510,3	677,0
	8	732,6	464,4	625,2
	9	737,3	471,1	623,1
	10	787,9	424,9	638,7
Wheat sampled manu-ally	1	480,7	378,2	452,3
	2	541,3	418,0	471,2
	3	485,5	468,8	478,1
	4	479,3	368,6	443,4
	5	474,4	406,7	455,1
	6	481,4	464,2	476,3
	7	457,4	389,0	432,3
	8	467,4	410,0	445,4
	9	462,6	445,8	451,9
	10	463,8	436,3	450,6
Wheat Sampled with non-auto-matic CS90	1	1062,5	470,1	824,6
	2	790,8	458,9	560,5
	3	760,2	760,2	760,2
	4	730,9	427,6	538,2
	5	846,5	390,8	599,1
	6	799,6	466,0	605,3
	7	765,5	117,6	487,3
	8	736,5	490,2	612,3
Wheat sampled with automatic CS90	1	633,9	449,2	525,2
	2	673,0	552,3	597,0
	3	627,6	483,7	571,0
	4	922,8	566,6	642,7
	5	611,4	483,3	546,1
	6	641,5	571,6	604,0
	7	663,0	552,9	606,0
	8	616,1	59,60	470,1

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Table 2 — Maximum, minimum and mean values for the analysis of the eight individual barley samples collected at Store 1

	Nitrogen (DM)	Moisture %	Volumic mass	Fines %	
Load 1				**Store**	**Project**
Maximum	1,77	14,4	69,1	4,6	2,9
Minimum	1,74	13,8	64,8	3,2	1,7
Mean	1,79	14,0	67,7	3,6	2,4
Load 2					
Maximum	1,77	14,4	69,0	3,9	2,6
Minimum	1,72	13,7	68,1	3,1	2,1
Mean	1,78	14,0	68,5	3,6	2,3
Load 3					
Maximum	1,75	14,1	69,5	4,3	3,2
Minimum	1,72	13,8	67,9	2,9	1,6
Mean	1,77	13,9	68,8	3,6	2,2
Load 4					
Maximum	1,76	14,3	70,0	3,6	2,8
Minimum	1,75	13,8	68,4	2,9	1,7
Mean	1,78	14,0	69,1	3,4	2,0
Load 5					
Maximum	1,76	14,3	69,7	5,2	2,9
Minimum	1,72	13,8	68,2	3,3	1,9
Mean	1,79	14,1	69,2	4,2	2,4
Load 6					
Maximum	1,75	14,1	69,1	5,6	3,3
Minimum	1,72	13,8	68,3	4,3	2,0
Mean	1,78	14,0	68,7	4,8	2,7
Load 7					
Maximum	1,75	14,3	69,2	6,4	2,9
Minimum	1,74	14,0	68,5	4,2	1,9
Mean	1,77	14,2	68,7	5,1	2,3
Load 8					
Maximum	1,76	15,0	69,5	5,5	3,4
Minimum	1,75	13,7	68,5	3,4	2,1
Mean	1,78	14,2	68,9	4,4	2,6
Load 9					
Maximum	1,76	14,1	69,4	4,4	3,4
Minimum	1,72	13,8	68,5	3,7	2,0
Mean	1,78	14,0	68,9	4,0	2,6
Load 10					
Maximum	1,74	14,5	69,0	5,4	3,5
Minimum	1,60	13,8	68,0	3,0	1,9
Mean	1,78	14,1	68,6	4,5	2,5

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Table 3 — Maximum, minimum and mean values for the analysis of the five individual wheat samples collected at Store 2

	Protein DM %	Moisture %	Hardness	Volumic mass	Fines %
Load 1					
Maximum	11,2	13,7	41,4	76,3	2,9
Minimum	10,8	13,4	37,1	72,9	1,8
Mean	11,0	13,5	39,0	75,1	2,4
Load 2					
Maximum	11,2	14,7	40,5	75,0	2,2
Minimum	10,9	13,8	34,1	72,1	1,7
Mean	11,0	14,2	38,0	73,9	1,9
Load 3					
Maximum	11,1	13,9	43,0	76,3	2,4
Minimum	10,7	13,7	35,6	75,2	1,9
Mean	10,9	13,7	38,0	75,7	2,2
Load 4					
Maximum	11,3	14,1	37,9	74,9	2,1
Minimum	10,7	13,8	31,6	73,7	1,7
Mean	11,0	13,9	35,4	74,4	1,8
Load 5					
Maximum	11,1	14,2	38,4	75,5	2,1
Minimum	10,6	13,5	34,3	73,3	1,6
Mean	10,9	13,9	36,8	74,8	1,9
Load 6					
Maximum	11,2	14,0	35,6	76,3	2,9
Minimum	10,8	13,3	28,7	74,8	2,0
Mean	11,0	13,6	33,0	75,8	2,4
Load 7					
Maximum	11,1	14,9	36,5	75,2	2,5
Minimum	10,5	14,3	29,3	74,4	1,7
Mean	10,8	14,6	32,4	74,9	2,1
Load 8					
Maximum	10,6	14,5	31,7	75,4	2,2
Minimum	10,2	14,3	24,1	74,5	1,8
Mean	10,4	14,4	28,6	75,0	2,0
Load 9					
Maximum	11,0	14,8	37,2	75,6	2,3
Minimum	9,9	14,5	16,5	73,8	1,9
Mean	10,6	14,7	29,2	74,6	2,1
Load 10					
Maximum	11,6	15,7	40,5	75,5	2,1
Minimum	10,9	14,6	16,6	73,5	1,4
Mean	11,2	15,0	33,7	74,8	1,8

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Table 4 — Maximum, minimum and mean values for the analysis of the eight individual wheat samples collected at Store 3

	Protein DM	Moisture %	Hardness	Volumic mass	Fines %
Load 1					
Maximum	14,0	14,3	62,7	82,6	1,4
Minimum	13,7	14,1	55,9	82,0	0,5
Mean	13,9	14,1	58,5	82,4	0,8
Load 2					
Maximum	14,0	14,1	59,5	82,5	1,3
Minimum	13,8	14,0	55,9	81,8	1,1
Mean	13,9	14,0	57,8	82,1	1,2
Load 3					
Maximum	14,2	14,0	58,4	82,6	1,4
Minimum	13,7	13,9	53,1	81,4	1,0
Mean	13,9	14,0	55,7	82,2	1,2
Load 4					
Maximum	14,1	14,0	59,5	82,9	1,2
Minimum	13,8	13,9	54,7	82,4	0,9
Mean	14,0	14,0	57,5	82,7	1,0
Load 5					
Maximum	14,3	14,1	77,3	83,2	1,2
Minimum	14,0	13,8	53,8	82,8	0,6
Mean	14,1	13,9	61,2	83,0	1,0
Load 6					
Maximum	14,1	14,1	59,5	83,0	1,2
Minimum	13,8	13,9	55,8	82,2	0,7
Mean	13,9	14,0	57,3	82,7	0,8
Load 7					
Maximum	13,9	14,1	58,7	82,8	1,2
Minimum	13,7	14,0	56,5	82,0	0,8
Mean	13,8	14,0	57,4	82,4	0,9
Load 8					
Maximum	13,9	14,0	58,9	82,9	1,3
Minimum	13,8	13,9	53,7	81,8	0,9
Mean	13,9	14,0	57,2	82,5	1,2

Table 5 — Maximum, minimum and mean values for the analysis of the eight individual wheat samples collected at Store 4

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The results of the store's own analysis, based on a composite sample made up from 8 individual probe samples, are given as Mill data.

	Protein (DM)	Moisture %	Hardness	Volumic mass	Fines %
Load 1					
Maximum	13,0	12,4	68,9	77,7	3,5
Minimum	12,7	12,1	61,9	76,5	1,9
Mean	12,8	12,3	65,2	77,0	2,63
Mill data	13,3	11,9	6	77,6	3,1
Load 2					
Maximum	11,8	14,1	40,5	76,8	4,2
Minimum	11,6	13,7	31,3	75	3,4
Mean	11,7	13,9	36,3	76,0	3,88
Mill data	11,8	14	2	76,8	4,9
Load 3					
Maximum	13,9	13,0	51,4	80,6	4,2
Minimum	13,4	12,6	42,2	79,5	3,4
Mean	13,7	12,7	47,0	80,1	3,88
Mill data	13,7	13,3	6	80,7	5,1
Load 4					
Maximum	12,4	13,4	46,2	79,7	1,9
Minimum	12,0	12,8	33,8	77,8	1
Mean	12,2	13,2	41,5	79,0	1,35
Mill data	12,6	13,6	2	79,5	2
Load 5					
Maximum	12,5	15,0	28,1	78,4	2,8
Minimum	12,1	14,7	21,5	75,9	1,8
Mean	12,3	14,9	24,50	77,3	2,23
Mill data	12,1	15	2	77,6	3,2
Load 6					
Maximum	12,5	12,8	48	78,4	2,1
Minimum	12,2	12,5	39,2	76,6	1,9
Mean	12,3	12,6	43,2	77,8	1,95
Mill data	12,5	12,8	2	79,1	3,2
Load 7					
Maximum	13,0	12,2	55,1	81,6	2,3
Minimum	12,5	12,0	41,2	80,1	2
Mean	12,7	12,1	47,2	81,0	2,14
Mill data	12,8	12,2	5	81,1	3,2
Load 8					
Maximum	11,3	13,2	38	77,5	2,2
Minimum	11,03	13,1	29	75,9	1,6
Mean	11,2	13,2	34,6	76,7	1,88
Mill data	11,4	13,4	2	77,6	2,8

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Table 6 — Maximum, minimum and mean values for the analysis of the samples taken by the storekeeper during intake at Store 3

Figures in brackets are the mean value of the 8 loads sampled coming out of store.

	Protein (DM)	Moisture %	Volumic mass	Fines %
Maximum	14,0 (14,1)	16,0 (14,1)	82,0 (82,8)	4,3 (1,3)
Minimum	10,9 (13,8)	12,1 (13,9)	74,1 (82,1)	0,1 (0,8)
Mean	12,5 (13,9)	14,1 (14,0)	78,1 (82,5)	2,2 (1,0)

A summary of the analyses of the Falling Number of some samples from Stores 2 and 3 are given in Table 7. Three replicates of each sample were tested and the mean values used in the calculation of maxima, minima and means. These data show that there was important variation between the values obtained from the individual samples. However, it must be appreciated that the variability (commonly, ±4 s to 6 s) between replicates of the same sample *i.e.* measurement variation, could account for up to 20 % of the variation between samples.

Table 7 — Results of falling number assessments on individual samples from some loads at stores 2 and 3

	Load				
Store 2	3	5	6	7	8
Maximum	229	226	220	245	205
Minimum	168	183	172	185	181
Mean	198	203	200	218	194
Store 3	5	6	9	10	
Maximum	183	164	193	186	
Minimum	152	131	163	122	
Mean	166	147	175	168	

The tests on the germinative capacity done on barley samples from Store 1 showed very little variability between samples with all values being 99 % or 100 %.

At store 4 (a flour mill), normal practice was to collect 8 samples with an automatic Samplex CS 90 probe, mix these samples and then divide the resulting composite sample to give an analytical sample. The results obtained by the mill using their method of sampling and analysis are given in Table 5. In most cases comparisons can be made but the mill used a different method of assessing Hardness so these results cannot be compared.

5.4.2.3 Estimating the sampling reliability

The results of the Monte Carlo simulations are given in Table 8 and show the improvement in sampling reliability gained from taking more samples. The use of 8 samples reflects the number of samples that most operators take when using a CS90 automated sampler to sample from trucks on intake.

The ranges for each quality measurement were selected on the basis that they represented relatively small variation about the mean value.

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Table 8 — Impact of taking greater number of samples on the confidence limits for a given level of variation about the mean value (1 sur 2)

Wheat Hagberg Falling Number (see Figure 4) Mean = 203 Maximum = 217 Minimum = 183 using a range of 10 seconds either side of mean (193-213) the following confidence limits apply *i.e.* the probability of this interval covering the mean	
2 samples	79 %
3 samples	88 %
5 samples	96 %
amples	99 %
Wheat Specific Weight Mean = 82,04 Maximum = 82,6 Minimum of 81,6 using a range of 0,4 kg either side of mean (81,64-82,44) the following confidence limits apply *i.e.* the probability of this interval covering the mean	
2 samples	77 %
3 samples	85 %
5 samples	94 %
8 samples	98 %
Wheat Moisture Content Mean = 13,93 Maximum = 14,06 Minimum = 13,87 using a range of 0,1 % either side of mean (13,83-14,03) the following confidence limits apply *i.e.* the probability of this interval covering the mean	
2 samples	69 %
3 samples	79 %
5 samples	90 %
8 samples	96 %
Wheat Protein Mean = 14,02 Maximum = 14,24 Minimum = 13,79 using a range of 0,1 % either side of mean (13,92-14,12) the following confidence limits apply *i.e.* the probability of this interval covering the mean	
2 samples	59 %
3 samples	69 %
5 samples	82 %
8 samples	91 %
Barley Screenings Mean = 2,04 Maximum = 2,8 Minimum = 1,7 using a range of 0,3 % either side of mean (1,74-2,34) the following confidence limits apply *i.e.* the probability of this interval covering the mean	
2 samples	67 %
3 samples	76 %
5 samples	87 %
8 samples	95 %
Barley Moisture Content Mean = 14,06 Maximum = 14,28 Minimum = 13,82 using a range of 0,1 % either side of mean (13,96-14,16) the following confidence limits apply *i.e.* the probability of this interval covering the mean	
2 samples	58 %
3 samples	70 %
5 samples	79 %
8 samples	89 %
Barley Nitrogen Mean = 1,77 Maximum = 1,78 Minimum = 1,72 using a range of 0,01 % either side of mean (1,76-1,78) the following confidence limits apply *i.e.* the probability of this interval covering the mean	

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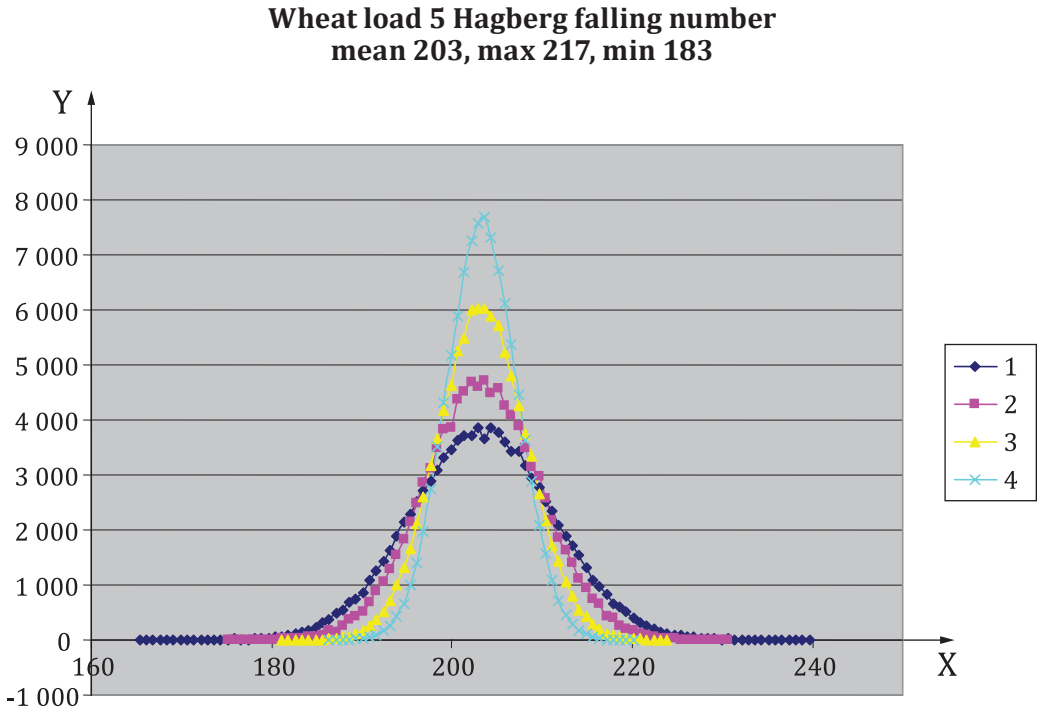
Table 8 *(continued)*

2 samples	52 %
3 samples	61 %
5 samples	71 %
8 samples	84 %

Figures 4 and 5 show two graphical representations of the results of the Monte Carlo simulations on reliability of results from using differing numbers of samples.



Key

X hagberg falling number

Y frequency

1 2 samples

2 3 samples

3 5 samples

4 8 samples

Figure 4 — Graphical representation of the impact of sampling intensity on reliability of result for Hagberg falling number in a lorry load of wheat

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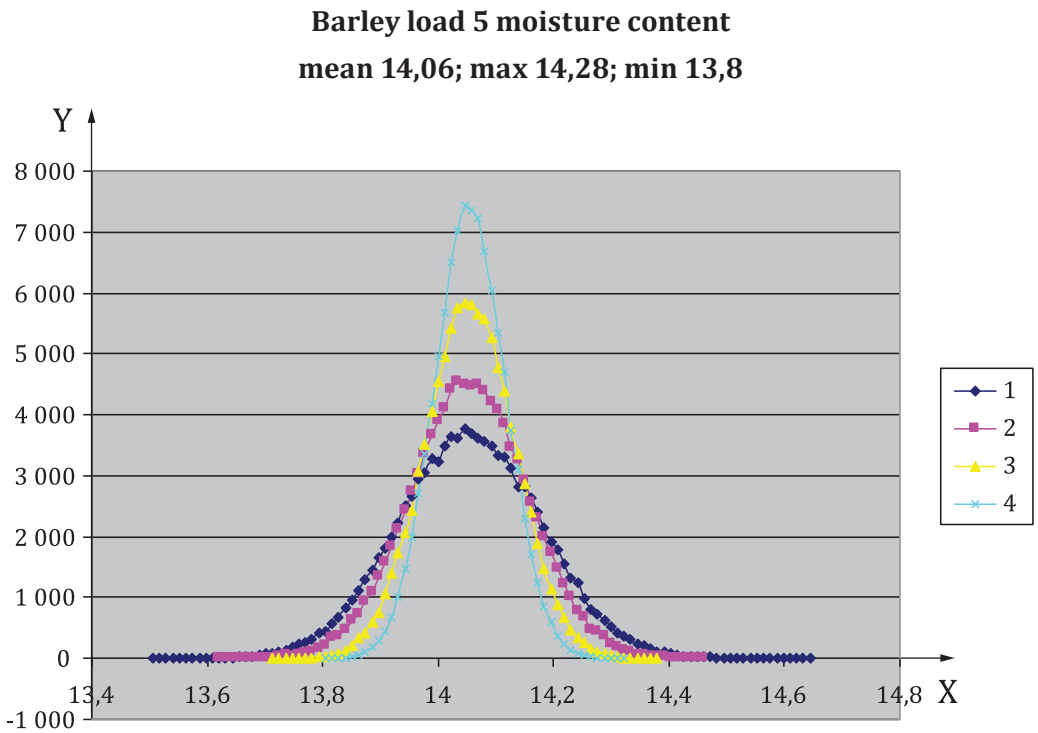

Key

X moisture content

Y frequency

1 2 samples

2 3 samples

3 5 samples

4 8 samples

Figure 5 — Graphical representation of the impact of sampling intensity on reliability of result for moisture content in a lorry load of barley

When the differences between the results of using a mean of individual sample measurements, a mean from a series of samples from a composite or the mean from a series of samples taken from a single large bulk sample were compared there were no statistically significant difference between them. On some occasions, the difference was positive whilst in others the difference was negative. The types of samples that showed the least difference (analysed by eye) were the mean of the eight individual samples and the mean of the 5 samples from the composite sample.

5.4.2.4 Discussion and conclusions

The collection of information on the approaches to sampling lorries under practical conditions suggested that there were wide differences in approach used by different stores. The Samplex CS90 was the equipment most commonly used although the exact method of use varied greatly. The next most common method was a manual spear but once again, the number and size of sample collected varied. The information collected supports the targeting of CS90 samplers and manual spearing during this project.

Guidelines for lorry sampling are specified in some contracts. These are all based directly on the ISO and BSI standards and specify the number and positions of sampling points for loads of certain weights. However, during the course of this work it became apparent that, in practice, with manual sampling it was impossible to observe these guidelines and to also observe HSE safety constraints and food safety regulation advise against an operator walking on the grain. Similar limitations applied to the

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use of the CS90. When using this machine, even if ideally placed, it is impossible to reach up 40 % of the load in modern bulk trailers without moving the lorries. It is not common practice to move vehicles to overcome this limitation and therefore the sampling is limited to a central portion of the trailer in most cases. The manufactures of the CS90 are aware of this limitation but point out that, whilst it is possible to re-design the machine to extend its reach to cover the entire load, this would require a much larger and far more expensive unit. These are important issues and need to be addressed by the British Standards Institute and those responsible for drawing up contracts.

5.4.2.4.1 Inherent variation in grain

All grain sampled during this work at stores 1, 2 and 3 had been in store for at least 8 months. The grain had originally come from a number of sources but would have been mixed to some extent when entering storage. It is also reasonable to expect that extremes in moisture content would have been much reduced during storage. This is confirmed by the reduction in the difference between the maximum and minimum values for moisture shown by comparing the intake sampling at Store 3 with the values obtained during the project when assessing the grain at out-loading. However, the results obtained indicate that some inherent variation in the grain is still present after bulks of grain from farms are mixed and stored at commercial stores. The variation in the quality of grain within in a single lorry load was sufficient to make the difference between the grain being rejected or accepted. For example, the moisture content of load 10 at store 2 varied sufficiently that a single sample could have permitted the end user to accept or reject given a contract level of 15 %. Similar important variation was apparent in the protein content and falling number values of samples from Store 3. The origins of the grain at store 4 are unknown but at least some loads are likely to have come directly from farm stores.

In the case of the two stores where the grain was sampled with a CS90, the variability between individual samples may have been enhanced by the large differences in sample weight; up to two-fold. However, large differences in sample weight did not occur at Store 2 where the grain was sampled manually yet variation in quality was still present between individual samples.

Another factor that could influence the apparent variation between samples is any change to the method of measurement. The assessment of this factor was not a direct part of this project. However, some observations on the effects of changes in the method of measurement on the results were obtained. Large difference in screenings were found in the same samples of barley (Store 1) depending on whether they were tested by the store staff or project staff (see Table 2). The mechanical shaker used at the store always produced a higher assessment of fine material (+ about 30 %) than the manual sieving used by the project staff. However, this is not an indication of variability in the grain because the difference between the 2 methods remained about the same. These findings are not surprising as the model and setting of the shaker and the age of the sieves can influence the results (C. Finch, Pneumac, pers. com.). In addition, the results from all samples of both wheat and barley collected from lorries and those from other parts of the Grain Sampling and Analysis (GSA) project, indicate that the distribution of fine material or screenings within a bulk or lorry-load of grain is variable and, as far as can be judged from results, random. As a result, the measurement of screenings is very likely to be subject to larger errors of prediction than other quality parameters. This may be worthy of more detailed research and perhaps should be born in mind when considering contractual obligations.

Tests done on other quality parameters throughout the GSA project on the Foss Infratec machines suggest that this instrument is relatively consistent. Where the same sample was put through the machine multiple times there was almost no variation in the results. The results obtained at Store 4 suggest that the values obtained by the project analysis were in general lower than those obtained by the mill analysis although the differences were mostly relatively small. It is possible that this trend was more a factor of analytical methods than sampling error. The largest difference between the two assessments was always in the estimation of screenings.

In general, the variation between samples taken from the same lorry-load was not of statistical significance. However, in many cases these differences were of great commercial importance. For example, with barley from Store 1 the screenings could vary by 1,5 % and the moisture by 1,4 % between samples from the same load. With milling wheat from store 3 the protein could vary by 0,5 % and the screenings by 1 % within a load. These data clearly demonstrate the dangers of basing a quality assessment of a lorry-load of grain on a single discrete sample irrespective of the method of collection.

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Statistical analysis of the data provided useful information. The amount of variability for each quality parameter within a load or a group of loads was calculated and the ability to predict this variability was assessed. Obviously, each quality parameter behaved differently but most was broadly similar except screenings where the variation was much greater. Once the variability was calculated, limits could be set for the accuracy of prediction (for example: ± 0,05 % for protein in wheat). The numbers of samples used has a fundamental influence on the ability to approach these limits. A single sample may only offer a 50 % chance that the result will be close to such limits. In practical terms, this means that when lorries are sampled with a single sample, in the event of a dispute, there is a 50/50 chance that re-sampling will give a different result.

The confidence of prediction rises as the number of samples used in the assessment rises and 8 samples/load would appear to give a good compromise between confidence in the results and workload. There was no relationship between quality prediction and the position from which a sample was collected: the variability appeared random. Therefore, the exact siting of the sample points is of much less importance than the number of sample points, although spreading the points evenly over the entire load should be preferred. Collecting multiple samples (preferably 8/load), mixing these thoroughly and then using a sub-sample for quality assessment would appear to be an effective approach to sampling lorry-loads of grain.

The results from this work confirm the earlier limited work on sampling lorries (HGCA Project Report 79) but it does not appear possible to implement the use of the methodology suggested in ISO or BSI Standards because of limitations in the current equipment. However, they provide the basis for a new approach to standards with a sound technical base and may allow some general codes of practice to be constructed.

5.4.2.4.2 Recommendations and protocol for lorry sampling

5.4.2.4.2.1 Recommendations

a) **Screenings**

Throughout the sampling component of the GSA Project, the error of prediction of screenings has been greater than other quality parameters. This difficulty does not seem to be reflected in commercial contracts. It is recommended that more work be done to establish better limits of assessment that could be used that would provide a fairer approach for both buyers and sellers of grain.

b) **National and International Standards**

The results of this work should be discussed with the appropriate Standards organisations with a view to producing revised standards that correctly reflect the limitations of equipment and safety concerns.

Code of Practice: consideration should be given, in cooperation with appropriate organisations, to the drawing up of guidelines for the effective sampling of loads of grain.

c) **Influence of method of measurement**

Consider the interchange of samples between the project and commercial laboratories to investigate the effects of different methods of measuring quality parameters of grain.

5.4.2.4.2.2 Protocol for collecting samples from lorry-loads of grain

These guidelines are intended to offer advice to any organisation that sample grain in lorries. They are not intended as dogmatic instructions that can be applied at every site and there may be a need for some flexibility in approach. However, if used, the guidelines will help to minimise sampling error and provide a consistent method of collecting samples from lorry-loads of grain.

a) **General**

— All equipment should be clean and in good working condition;

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— Operators should be familiar with the equipment and have an understanding of the problems of sampling grain;

— Safety should always be the first consideration in any sampling operation.

b) **Taking manual samples:**

— Use a manual spear that is at least 1,5 m long;

— Use a spear that will collect multiple sub-samples from different depths;

— Collect about 500 g/sample point. This may require 2 insertions/sample point;

— Collect samples from at least 5 (preferably 8) sample points/load. These points should be spread as widely as possible across the load without risk to the operator. In some case, it may be appropriate to move the lorry to give better access;

— Add the samples together to form a composite sample representing the load;

— Mix the composite sample thoroughly;

— Divide the composite sample with a sample divider. If this is not available the sample should be divided by coning and quartering to give a working sample. Failure to use a sample divider or to cone and quarter the sample is likely to increase the error in the measurements (particularly screenings);

— It may be appropriate to retain part of the composite sample in case of any dispute over the grain quality.

c) **Taking samples with a remote sampling probe**

— Where the amount of grain collected per insertion can be adjusted, it may be appropriate to set this to maximum. This will help to overcome the collection of small samples when the spear is inserted into troughs in the load;

— Try to ensure that the lorry is positioned with the sampler about half way along the trailer so that it can access as much of the load as possible;

— If the sampler has an automatic programme, choose the option that collects 8 samples from different points;

— If the probe is controlled manually, collect 8 samples/load from different points spread as far apart as possible over the load;

— Mix the composite sample thoroughly and then divide using a sample divider or by coning and quartering. Failure to use a sample divider or to cone and quarter the sample is likely to increase the error in the measurements (particularly screening);

— It may be appropriate to retain part of the composite sample in case of any dispute over the grain quality.

6 Study n°2: extract from "Sampling grain in static and flowing condition – alternatives to the regulatory protocol"

6.1 General

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6.2 Context

6.2.1 Regulatory aspect

Regulation has been published at European level concerning sampling and methods for analyzing cereals and cereal products, within the framework of assessing average batch mycotoxin content: regulation n° 401/2006 of 23/2/2006 (Official Journal of 9/3/06).

The regulation concerns official controls. Nevertheless, if there is a too large gap between a protocol applied by the official bodies in charge of regulatory checking and those implemented by the cereal industry operators, this might result in different evaluations of the average content of the batches of cereals. This situation is a potential source of disputes. Commercial operators are directly concerned with these regulatory protocols insofar as they are entrusted with some official analyses (*e.g*. within the framework of Community intervention).

Furthermore, attempts to take the “contaminants” issue into account in the "Intervention" procedure, in particular for the application of **(EC) 856/2005** regulation, are confronted by certain concrete limits in implementing methods specified for sampling, due to the characteristics specific to big cereal batches concerned and in particular their size.

6.2.2 Normative aspect

Three main international sampling standards were available at the time of the studies:

— ISO 24333;

— ISO 542:1999;

— ISO 6497:2005.

These standards only deal with the evaluation of “uniformly” distributed characteristics ("homogeneous") such as protein or moisture.

ISO 24333 is the only one proposing sampling protocols suited to contaminants spread in a heterogeneous manner, and in particular in the field of mycotoxins such as *Fusarium* toxins.

NOTE The two standards ISO 6644:2002 and ISO 13690:1999 were withdrawn at the publication of ISO 24333.

6.3 Studies conducted and objectives

Two studies were conducted: the first in 2004 to 2005 (“study A”) and the second in 2006 to 2007 (“study B”).

6.3.1 Study A

The purpose of this study was respectively:

a) to compare 3 protocols with the aim of estimating the possible differences between:

— a "regulatory" protocol whose sampling method corresponds to the one set out in EU regulation n° 401/2006[2];

— a "normative" protocol whose sampling method is adapted to certain situations experienced by cereal operators (e.g. intervention scheme);

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— a “routine” protocol whose sampling method is adapted to “day to day” control by cereal operators (e.g. intervention scheme).

For the 3 protocols, an aggregate sample was made from the individual samples and then analysed to determine an average content of the batch and to assess the related accuracy.

b) to evaluate the heterogeneity of 3 silos for the analysis of DON; of 2 silos for the analysis of nitrogen and falling number; of 1 silo for the analysis of moisture. For each of these analysis/silo, the 100 samples were individually analysed.

6.3.2 Study B

This study was conducted on large storage silos, on flowing of grains of corn, contaminated by Fusarium toxins, mainly fumonisins, but also deoxynivalenol and zearalenone.

The aim of this study B is to determine relationships between the average mycotoxin content and the variance of silos in order to estimate, for a given number of samples and for a given level of contamination, the accuracy that can be expected.

A part of this study was also devoted to assessing the impact of reducing the size of the laboratory sample.

6.4 Study A: silos and lorries of wheat and corn – Fusariotoxins and quality assessment

6.4.1 Organising field tests

6.4.1.1 Conducting tests

6.4.1.1.1 Setting up the tests

The tests were conducted on volunteer sites. A questionnaire was prepared and circulated in order to identify the interesting sites.

Furthermore, in order to ensure a prompt reaction when a batch was identified, and to ensure that the tests conducted in a reliable and rigorous manner, a single person was entrusted with conducting all of the samplings carried out in the field.

The same method was applied to similar situations, as far as possible, with the same equipment being used for all the trials: manual probes, divider and grinders. The automatic probes varied from a site to another.

6.4.1.1.2 Methods corresponding to the protocols tested

2 series of tests were conducted:

— the 1st series consisted of testing 3 sampling plans: the protocol corresponding to regulation n° 401/2006 for fusarium toxins, the "normative" protocol drafted by the "standardisation” working group (ISO 24333) and the “routine” protocol with less incremental samples per unit than the 2 others.

The grain sampling situations were as follows:

— flowing from silos (transferring the contents of one silo to another one, discharge and Redler samplings, or train discharge);

— static by sampling from lorries;

— static by sampling from flat or vertical silos.

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The aim was to see whether the alternatives protocol correctly estimates the average content of a batch in the same way as the regulatory protocol, taking into account some experimental errors. The regulatory protocol is used here as a reference. The characteristics used for estimation are the DON content, the protein content and the Hagberg Falling number for wheat. For maize, the content values taken into account are: DON, protein content, promatest and zearalenone.

— The aim of the 2nd series was to evaluate silo heterogeneity in order to be able to estimate the relationship between the confidence interval of the average content of the silo and the number of incremental samples. Furthermore, resultant modelling enables to evaluate the risk taken if the batch is accepted on the basis of the number of incremental samples chosen.

The heterogeneity tests were conducted on silos of 500 t and for each, 100 elementary samples were taken and individually analysed: 3 silos for the analysis of DON; 2 silos for the analysis of nitrogen and Falling number; 1 silo for the analysis of moisture. The batches tested weighed approximately 500 t. Heterogeneity was only tested on wheat batches.

Table 9 — Characteristics of the tests conducted

Tests of sampling plans : regulation / standard / routine						
				regulatory method	normative method	routine method
flowing grains (by 500 t)	Type of grains	nb essais				
	wheat	1	sampling equipment	scoop	scoop	scoop
			number of increments	100/500T	25/500T	10/500T
	corn	4	mass of increment (kg)	0,3 à 1,6	0,3 à 1,8	0,3 à 1,9
			mass of aggregate sample (kg)	32 à 165	8 à 44	3 à 19
			mass of laboratory sample (kg)	9,5 à 9,8	8 à 10,6	3 à 10,4
			division of the aggregate sample	Boerner divider	Boerner divider	
Static lorries						
	wheat	6	sampling equipment	manual or automatic sampling equipment	automatic sampling equipment	automatic sampling equipment
			number of increments	100	10	3
	corn	8	mass of increment (kg)	0,4 à 1,5	0,4 à 1,5	0,3 à 1,9
			mass of aggregate sample (kg)	43 à 150	3,8 à 15	1,2 à 4,2
			mass of laboratory sample (kg)	9,3 à 10,9	3,8 à 10,9	1,2 à 4,2
			division of the aggregate sample	Boerner divider	Boerner divider	Boerner divider
Flat or vertical silos (by 500 t or by batch)						
	wheat	3	sampling equipment	manual sampling equipment or probes by aspiration	manual sampling equipment or probes by aspiration	manual sampling equipment or probes by aspiration
			number of increments	100	50	10
			mass of increment (kg)	1,9 à 2,7	1,9 à 2,7	2,2 à 3,0
			mass of aggregate sample (kg)	180 à 265	95 à 140	22 à 30
			mass of laboratory sample (kg)	11	10,3	10,7
			division of the aggregate sample	Boerner divider	Boerner divider	Boerner divider
Tests d'héréogénéité						
Test of Heterogeneity on 2 flat silos and 1 verical silo	type de produits	nb essais				

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Table 9 *(continued)*

Tests of sampling plans : regulation / standard / routine						
				regulatory method	normative method	routine method
	wheat	3	sampling equipment	probes by aspiration		
			number of increments	100		
			mass of increment (kg)	1,8 à 2,7		
			mass of aggregate sample (kg)	180 à 265		
			mass of laboratory sample (kg)	1,8 à 2,7		
Tests realised 2005/09/20						

6.4.1.1.3 Description of the tests conducted

— 1st series → 25 tests were conducted on:

  — flowing grain: manual sampling equipment; 4 in silos - 3 maize, 1 wheat, from 500 t sections; 1 train –maize. The samples were taken at regular intervals in accordance with the flow and the number of samplings required;

  — static grains: manual or automatic sampling equipment; 14 lorries (6 wheat, 8 maize), 3 types of probe. In 10 cases, a manual sampling was used; In 7 cases, a mechanical sampling was used; In 3 cases, the manual sampling was compared to the mechanical sampling;

  — 2 vertical silos and 1 horizontal silo: on wheat; the samples were taken from 500 t batches using mechanical probes (suction-operated probes).

The aggregate samples are obtained by grouping together the individual samples corresponding to each sampling situation and each method → for each situation; there are thus two aggregate samples (regulatory method – normative method). The aggregate samples obtained were homogenized and then divided in order to obtain a 10 kg laboratory sample. Aggregate samples of less than 10 kg were just homogenized and then ground (Labormill grinders).

The diagrams below show sampling examples taken from lorries.

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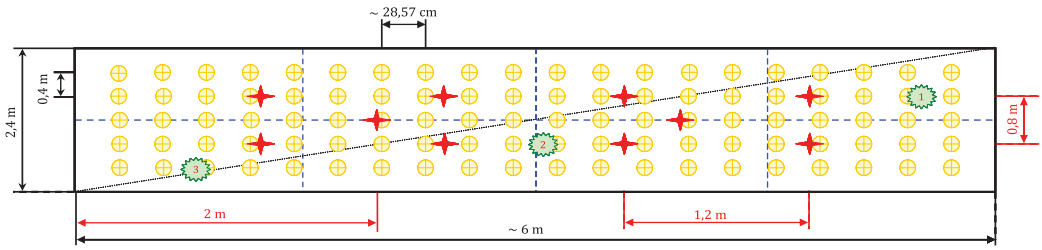

Key

:100 points of sampling

:10 points of sampling

:3 points of sampling randomly distributed

Figure 6 — Example of sampling from lorries

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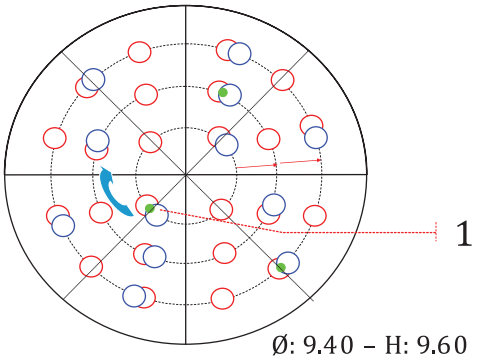

Figure 7 — Example of sampling from silo

Figure 8 — Boerner divider

Figure 9 — Example of sampling equipment

Key

○	24 places of sampling
○	12 places of sampling
●	3 places of sampling
1	1st sampling

— 2nd series: heterogeneity tests → 3 trials were conducted on 2 horizontal silos and 1 vertical silo. Grids were drawn on the top of the silos in order to ensure a consistent sample across the whole batch sampled. Each 100 elementary sample was individually homogenized and ground before being analysed.

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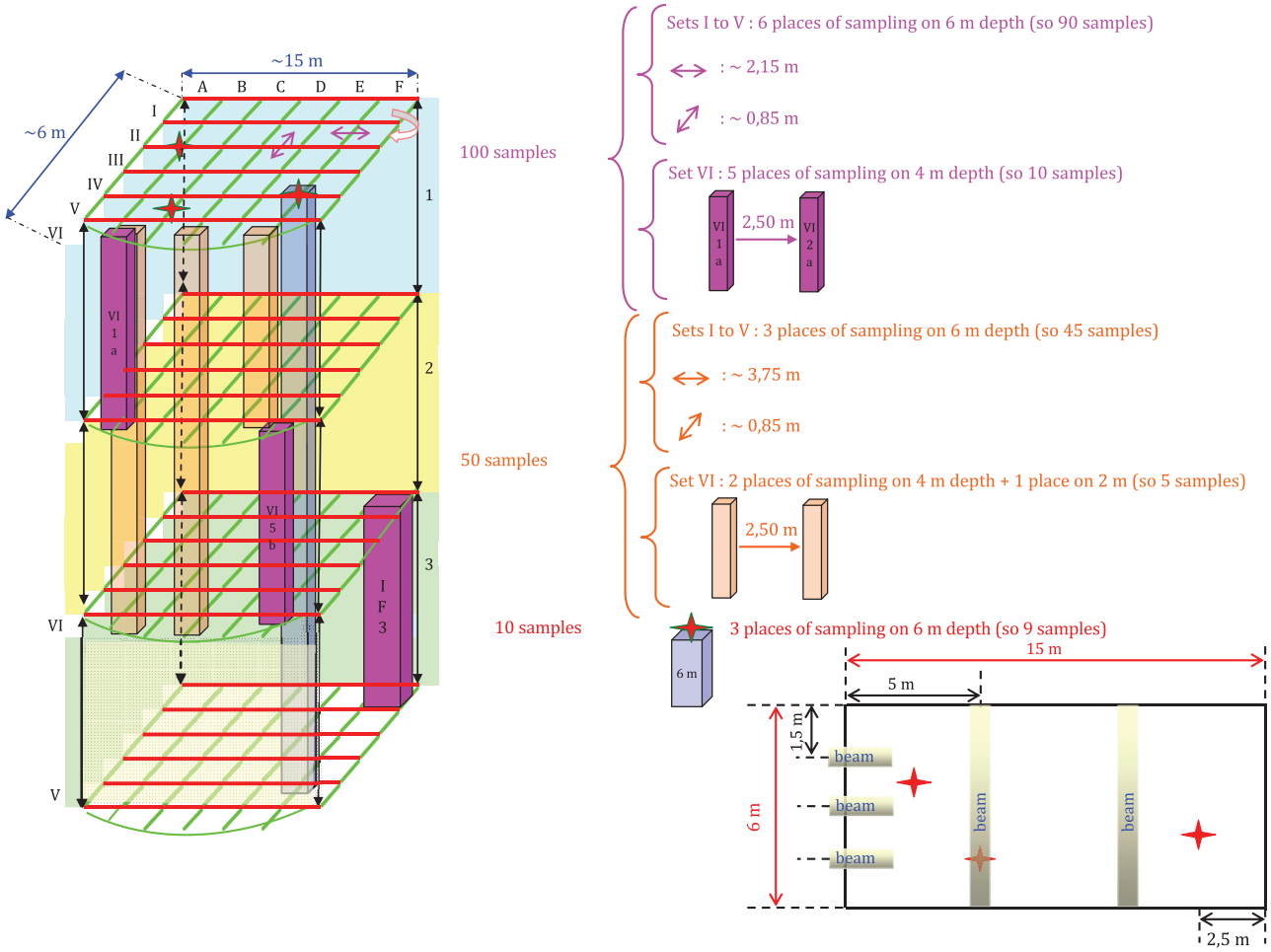

Figure 10 — Illustration of sampling from silos

— Table 10 summarises the different situations and sampling equipment.

— Tables 11 to 13 summarise the quantities sampled, homogenized, divided and ground, as well as the corresponding necessary times.

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