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Foreword

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This document was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 4, *Cereals and pulses*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 338, *Cereal and cereal products*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

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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.

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.

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.

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.

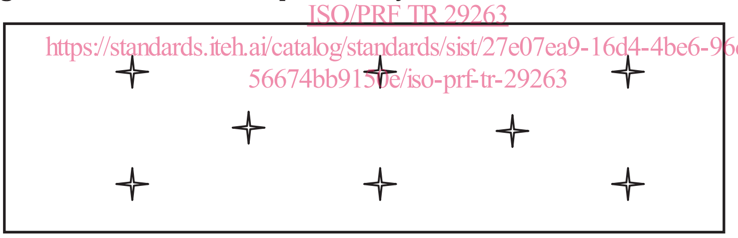

Figure 1 — Arrangement of sampling points used by CS90 sampler

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.

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.

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 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.

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 manually	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-automatic 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

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