HGCA

PROJECT REPORT No. 34

AN ASSESSMENT OF METHODS OF SAMPLING BULK GRAIN

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AN ASSESSMENT OF METHODS OF SAMPLING BULK GRAIN

by

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Summary

Removal and examination of samples of grain is the most widely used method of measuring quality parameters. However, there are few research data to support methods currently used or to quantify errors that may occur. Many variables, such as the equipment, operator or the type of grain storage, may affect the make-up of samples and, therefore, the results obtained.

A series of experiments was done to determine the effectiveness of various sampling methods at detecting insects in bulk grain and to assess the influence of these different methods on the results of quality measurements. It proved almost impossible to detect insects at densities below about 5/kg using commercial rates of sampling. There were large errors in predicting some quality parameters, such as total impurities and broken grains, but quality prediction was not affected by sampling equipment. Statistical analysis confirmed that the accuracy of prediction was related to both the number of samples from a single lot and the number of individual lots that were examined. The best method of assessing the quality of a bulk would appear to be to pool the results from the assessment of the individual lots that go to make up the bulk.

A series of recommendations is made for further research and development.

AN ASSESSMENT OF METHODS OF SAMPLING BULK GRAIN

1. General Introduction

1.1 Background to grain sampling

Removal and examination of samples is the standard method of establishing the quality of a batch of grain. The same technique is also used to determine the suitability of grain in store for further storage. Despite the obvious importance of the results yielded by sampling, only limited research seems to have been conducted in the U.K. on the validity of the methods of collecting samples which are currently in use. Investigations into grain sampling have been carried out in a number of other countries, notably U.S.A. and Australia, but the conditions, commodities and commercial requirements that influenced these experiments limit their value in the context of the U.K.

Originally, grain was stored and transported largely in bags. This system lent itself to a simple approach to sampling that could easily be defined in statistical terms: a consignment could be broken down into a number of standard units (1 bag). However, bulk grain, even in relatively small lots, is much less easy to categorise. For example, samples have to be collected at points over a three dimensional matrix. Large bulks or rapid flows of grain along conveyers, present even greater problems.

Despite the above problems, some methods at least are well defined: the method recommended for the examination of grain offered into intervention is laid down in the British Standard BS 4510, which is the same as International Standard IS 950. Another example of a standard method is contained in the GAFTA contract. This latter method gives some general indication on sampling and the handling of samples after collection, but does not specify the equipment to be used or take full account of the size of the lot of grain being sampled.

In practice, many sampling procedures are not subject to any official control. For example, for many years the method used by the Ministry of Agriculture, Fisheries and Food to confirm some aspects of the phytosanitary status of export grain, did not specify how the sample was to be collected. This situation has now changed and the BS method is specified for lorry-loads and a similar rate of sampling is used for flows of grain coming from silos. However, within the U.K., in the majority of cases where grain is sampled, the methods or equipment to be used are either not specified or poorly defined.

After collection, grain samples are assessed for a range of quality parameters that may include moisture content, hectolitre weight (specific weight), germinative energy, protein content or total nitrogen, amount of non-grain contamination, broken grains, dust or fine materials and the presence of specific contaminants such as insects, mites, weed seeds and ergot. Even the methods of determining these quality parameters are not standard throughout the cereal industry, further adding to the potential confusion that may occur when assessing grain quality.

1.2 Objectives of sampling

The objectives of sampling are usually, firstly to confirm that a specific lot of grain conforms to certain quality characteristics. This may allow the buyer to reject or divert sub-standard batches, or it may allow the sorting of a lot into a series of batches of ranging quality. Secondly, sampling may be carried out in an attempt to exclude unacceptable contaminants such as insects, or grain with a moisture content above a certain level.

1.3 Sampling attributes and variables

The characteristics measured during quality assessment can be divided into two types: attributes or variables. Attributes are characteristics that are or are not possessed; for example, the grain is wheat or barley, or it is or is not infested. Variables are characteristics that can vary in a stepless range; for example, moisture or protein content. In general, most quality characteristics that are measured, are variables. Even infestation may be regarded as a variable if quantified in terms of numbers of insects per kilogram of grain.

The characteristics of grain can be further divided into those that are characters of the grain and those that are formed by contaminants of one sort or another. Grain-related characters may or may not be distributed evenly through a lot of grain, depending on the size of the lot and its make up. Contaminants, such as insects, fine material and ergot, may or may not be distributed in a random manner but are very unlikely to be evenly distributed. Such contaminants are most likely to follow a random but clumped distribution. This poses an immediate problem for sampling for a single sample cannot accurately represent features that follow a clumped distribution.

Modern approaches to quality control have addressed the problem of establishing the attributes and variability of a production run. The methods would appear to have some application to grain sampling, particularly in respect of determining the number and size of samples needed to establish a particular quality characteristic. However, basic data are needed in order to investigate the extent to which these approaches can be applied to grain sampling.

1.4 Methods of sampling grain

The lack of standardisation in grain sampling mentioned earlier makes it difficult to draw up a simple plan for the assessment of sampling methods. In the U.K. at least three different types of equipment are used to collect grain samples and there are no data to indicate if the samples collected by different equipment are equivalent. Indeed, it is almost impossible to collect samples from identical positions and of the same weight using, for example, a gravity spear and a compartmented spear. The differences between samples collected by the two types of spear may or may not affect the results obtained when the samples are examined.

1.5 Variables associated with collection of samples

In practice, a number of variables may have a significant effect on the accuracy and validity of the result obtained when sampling grain. These variables include:-

a) The operator

Operator variation may include failure to follow instructions and failure to be consistent. This can be minimised by having standard operating procedures and by ensuring operators are fully trained.

b) The sampling equipment

The different types of spear will be described later. The type of spear used will affect the size of sample that can be collected and the parts of the bulk that can be sampled. For example, only relatively small samples can be collected down to a maximum depth of about 3m using gravity or compartmented spears. Vacuum samplers, however, are able to collect much larger samples and can often reach down to depths of 10m, but they require a power supply and may be difficult to manoeuvre across large areas of grain. In addition, different types of equipment may allow different operator errors to occur.

Gravity and compartmented spears also tend to mix the grain each time they are inserted. Some grains from the surface layers are dragged down to the point from which the sample is removed (see Fig. 1).

c) The condition of the grain

Grain to be sampled may be in the form of a small, relatively homogeneous lot, or a large bulk made up from grain from a wide variety of sources. In many cases, the way in which the grain is stored or transported can affect results and should, therefore, influence sampling procedures. For example, the depth of grain in a store should influence the type of equipment used and lorry loads will require a different approach than barge or ship loads.

These variables may help to account for the lack of standardisation in grain sampling in the U.K. They also provide ample justification for undertaking the scientific study into grain sampling reported below.

2. **Experimental work**

2.1 Objectives of the work

The principal objective was to compare the results obtained when samples were collected using three types of equipment. In addition, more detailed information was sought on the detection and estimation of insect populations. These objectives are not completely straightforward and could not be met in a single experiment. For example, the estimation of a variable such as moisture content is very different from determining an attribute such as whether or not grain is infested. Therefore, the results presented in this report were derived from a series of separate experiments, all performed under conditions that approached practical, commercial storage practice.

The first experiment was aimed at detecting insects in grain. This work did not use funds from the grant given by the H-GCA for this project, but the results are sufficiently relevant to warrant inclusion.

The second experiment compared the results obtained by examining samples collected using three types of sample spear. Multiple assessments were carried out on a batch of wheat and a single confirmatory assessment was carried out on a batch of barley.

2.2 Experimental detail

A. Detection and quantification of insects

These experiments used three 20-tonne bulks of English wheat. The grain was held in metal bins measuring about 3x3x3m and had a moisture content of about 15%. All the grain originated from the same source and was considered to be relatively homogeneous.

Two markers were used for the sampling assessment: dead adult beetles of the species *Oryzaephilus surinamensis* and *Sitophilus granarius*, and grains of wheat dyed blue. Each of the markers was added at the rates of 1/5kg, 1/kg and 5/kg as evenly as possible as each 20 tonne lot was turned between bins. Dead insects were used to avoid the unknown dynamic effects on distribution caused by insect movement. The same bulks of grain were used at each rate of inclusion so that the insects and dyed grains were added first at 1/5kg, the grain sampled, and then the grain was turned between bins and insects and dyed grains added at 1/kg. After further sampling, the grain was turned again and insects and dyed grains added at 5/kg. The rates of inclusion were, therefore, additive. In practice, the movement of the grain between bins may well have broken up many of the dead insects. However, the dyed grains should have been unaffected.

Two types of spear were used: a gravity spear, collecting about 250g of grain from a relatively discrete part of the bulk (see Fig. 2a), and a 1.8m long, compartmented spear with 11 compartments collecting about 300g of grain from 11 points per insertion (see Fig. 2b). Samples were collected with each spear at each of three points in the three bins.

After collection, the samples were sieved over a 2mm mesh to remove the insects, which were then counted. The sieved grain was spread on a tray and examined for dyed grains. A full account of this experiment, together with details of confirmatory work carried out by French scientists was presented at the 5th International Working Congress of Stored Product Protection, Bordeaux, 1990 (Wilkin and Fleurat-Lessard. The detection of insects in bulk grain. Conference Proceedings, In Press).

B. Comparison of sampling devices

The work was divided into two parts with a major sampling assessment being carried out on three occasions, followed by a repeat assessment carried out in the field using the same methods to provide confirmatory data. This work provided a large number of comparative data points that could be subjected to statistical analysis.

The first part of the work used three, 20-tonne lots of English wheat contained in the same metal bins as described in section 2.2A. The grain came from a single bulk but each bin was filled from a different lorry load.

Collection of samples:- Three devices were used: a gravity spear (see Fig. 2a), a compartmented spear (see Fig. 2b) and a vacuum sampler (see Fig. 2c). All equipment was standard and representative of the type of equipment in current use to collect grain samples in the U.K. None of the samplers were supplied with detailed instructions on their use but staff collecting samples were asked to follow a standard procedure that had been drawn up by the Central Science Laboratory over a period of time. Details of these procedures are given in Appendix 1.

The collection procedures varied with each sampler, but followed the methods likely to be used in commercial practice. Samples were collected with each device at three points across the surface of the grain. The gravity spear was used to collect sub-samples from the surface layer and at depths of 1m and 2m, which were combined into a single sample. The compartmented spear was inserted to its full depth and then opened and the grain from all compartments combined to give a single sample. Three samples from each point were collected and mixed to give a single composite sample of sufficient size for examination. The vacuum sampler used a 1.5cm diameter plastic probe and this was allowed to descend from the top to the bottom of the grain bulk largely under its own weight and the action of suction. The motor was stopped as soon as the probe reached the bottom of the bin and the grain collected was used as the sample.

The above techniques gave three samples per bin per device and the whole exercise was repeated on two further occasions with the same bins of grain. No changes were made to the grain or the sampling devices between the three occasions so that the results from the assessment of all samples could be used in the statistical analysis. A total of 27 samples for each spear was used in the statistical analysis.

Exactly the same technique of collecting samples was employed at a field site holding about 1000 tonnes of malting barley.

However, in this case, only a single set of samples for each device were collected to provide validation data for the laboratory results.

Examination of samples:- Five characteristics were measured on most samples: total weight, specific weight, moisture content, broken grains and total impurities. The definitions were those used in the specification of intervention quality. The methods of examination were also those specified in the intervention regulations. In addition, 100-grain weight was measured on samples from one exercise.

The staff carrying out the examinations of samples were given training in the techniques at a government-owned intervention store under the supervision of an H-GCA Regional Cereals Officer. The definitions of the quality characteristics and details of the methods of determination are given in Appendix 2.

All samples from each exercise were examined consecutively. The recommended intervention intake procedures were followed, so that the samples were weighed, the specific weight determined, then sieved and the fractions weighed or examined as appropriate. The 100-grain weight and moisture content were determined using the sieved grain.

Determination of specific weight using a chondrometer, required a sample with a mass of greater than 1kg. This was achieved with every sample collected with the gravity spear or compartmented spear except one. However, it is more difficult to control the weight of grain collected by a vacuum sampler and on six occasions the sample collected was too small to allow the specific weight to be determined.

Analysis of data:- In addition to presenting the data as maxima, minima and means, a substantial amount of statistical analysis was done. For this, results from the laboratory trial with wheat were pooled but those from the field assessment of barley were analysed separately. The statistical package GENSTAT5 was used throughout.

The mean, variance and standard deviation of the results for each device and measurement were examined for patterns of variation due to the device or statistical heteroscedasticity. Where variation was apparent, a random effects analysis of variance was carried out for each measurement in order to determine the variation attributed to differences between exercises, positions in the bin and sampling devices. This allowed values to be attributed to differences caused by bin-to-bin or position-to-position variation and comparisons to be made between these and variation caused by the different devices used.

2.3 Results

A. Detection of insects

A large number of samples (27 for the compartmented spear and 81 for the gravity spear) were collected and examined during this experiment at each rate of inclusion of the markers. This rate of sampling is, of course, far in excess of the numbers of samples used in commercial practice. However, they do allow the data to be assessed in a number of ways.

The results of the examination of the samples removed from the three bins, at each of the inclusion rates, are given in Table 1 (see page 28). The results for sample weight, fine material, insects and dyed grains, are presented as means, with standard deviations, for all samples from the three bins.

If insects in grain are considered as a variable then the results of a sampling exercise would be expressed as numbers per kg so that the level of infestation could be estimated. Therefore, the level of insects and dyed grains per kg were calculated from the results and are presented in Table 2 (see page 29). It should be noted that results do not separate the two species of insects, so that the theoretical level should be twice that for dyed grains.

Alternatively, insects can be considered as an attribute, when the objective would be to classify grain as infested or not. Therefore, the number of samples that contained one or more insects at each inclusion level are shown in Table 3 (see page 29).

B. Comparison of sampling devices

The mean values and the standard deviations for all the quality parameters, as shown by the three different sampling devices when used to sample the wheat, are given in Table 4 (see page 30). The results obtained when sampling barley, are shown in Table 5 (see page 32).

These results indicate the degree of variation in the methods of sampling and sample assessment. They also supply a good indication of the mean values of the quality parameters for the grain used in the work. The means and standard deviations obtained for each sampling device, when all results for the wheat and barley were pooled, are shown in Table 6 (see page 33). The true variation of each individual component was assessed by removing the influence of the others, to give the typical, calculated errors, of determination (Table 7, see page 34). This indicates which individual features of the sampling exercise led to errors and the relative size of the errors. Further calculations were made so that the effects of different numbers of samples, and the number of lots sampled, on the likely error could be shown. These data, which show the size of error and relate closely to the practical assessment of lorry-loads of grain, are shown in Figs 3, 4, 5, 6, and 7. The large variability for quality factors such as Broken Grains and 100-

Grain Weight, in what was thought to be relatively homogeneous masses of grain, is rather surprising. However, no reasons can be suggested that would indicate that these results are not typical. Indeed, the data for wheat are supported by the results from the field assessment of barley.

Taking account of the standard deviations about the means, there are few significant differences in the quality assessments produced by the three sampling devices. Exceptionally, with barley, the vacuum sampler gave results for total impurities that were significantly different from and were more variable than those obtained with the other two devices. There were also differences between the devices in terms of variability around the mean for specific aspects of quality. For example, results for moisture content obtained using the gravity spear were more variable than those from the other two devices. A likely explanation for this is that the gravity spear produces a more discrete sample that would be more likely to show up variation in the moisture content due to stratification effects. However, the size of the error is small in relation to the measurement, so is unlikely to be of practical importance.

2.4 Discussion and Conclusions

A. Detection of insects

The small standard deviations for weights of samples (Table 1) shows that the method of collection was consistent and reliable. However, the standard deviations for the fine material were large, indicating that there was much variability within the bulk of grain or with the method of examination, or both. The commercial implications of this are that a large number of samples may have to be taken from a single bulk in order to obtain a representative assessment of the level of fine material.

No consistent differences between recoveries of dyed grains and dead insects were apparent. This would suggest that, as the dyed grains were very likely to have behaved in the same way as the rest of the grain during turning between bins, the distribution of the dead insects should also have been relatively even.

Taking into account the very large standard deviations, the results from the inclusion rate of 1/5 kg indicate that the sampling frequency used was too low to give reliable detection of either dyed grains or insects. Significantly, it should be noted that the sampling frequency used, of about 4kg/20 tonnes of grain, is four times the industry norm. The results at the inclusion rate of 1/kg were little better. At neither level do the results give any reliable indication of the population density of insects.

At the rate of five insects or dyed grains/kg the results suggest that there was a good chance (about 50%) of detecting insects or dyed grains with a single sample. However, the standard deviations were still very large. When all the results of the analysis of the sample assessments are pooled (Table 3), it is possible to make predictions about the ability of sampling methods to detect insects or dyed grains. The conclusion must be that infestations below five insects/kg will not be detected with any reliability by collecting and examining samples at a normal, practical rate. Even at the inclusion rate of 5/kg, only one in two samples contained insects. Therefore, when insects are detected by collection and examination of samples, it is probably an indication of an infestation rate of more than 5/kg. It would also appear that the withdrawal and examination of samples is very unlikely to give a good indication of the level of infestation (Table 2). This result must have implications for various research topics on pest detection, as well as adding to the problems of storekeepers.

The commercial implications of these results are important. The numbers of samples taken during the experiments were very large in relation to the amount of grain, yet the standard errors for all data, except the weights of samples, were very large. The following key points would appear to arise from the work:-

Firstly, the large standard deviation in the range of fine material recorded from relatively small bulks of supposedly homogeneous grain illustrates the inability of any sampling procedure to give a precise estimate of this quality parameter.

Secondly, the detection of insects by sampling would only appear to be reliable when the insects are present at relatively high densities. In practice, it may be possible to sample selectively, using knowledge of insect behaviour and the physical conditions within a bulk, and to concentrate sampling on areas most likely to contain insects. However, it is unlikely that these measures can be applied to bulk grain in transit that has been recently loaded into lorries, etc. In this case, the only way of increasing the chance of detecting pests is to maximise the number of samples examined or, more particularly, the amount of grain examined. In any event, the detection of a single insect in a spear sample must be considered to indicate the presence of an infestation of five or more insects/kg.

B. Comparison of sampling devices

When assessing grain for the range of quality characteristics used to determine suitability for intervention storage, little difference in results could be attributed to variation caused by the three different devices tested. No device gave consistently high or low results across the quality parameters and no device was consistently more or less variable than the others. However, with barley, the samples collected with the vacuum sampler gave much more variable results for Total Impurities than either of the other devices. This was not found for the wheat, so may be an artefact associated with that specific batch of barley, However, this point is in need of more investigation.

Significant variation in all the quality parameters was found between bins, between positions in each bin, and between samples, indicating that the grain was not homogeneous. This was surprising as all three bins were supplied from the same source at the same time and the grain had remained in the bins for many months. By chance, each bin represented one lorry-load, so the variation might be typical of that experienced when sampling lorries delivering grain to a store from a single source. It must be assumed that the potential for variation must be much larger when several sources are involved. The differences between exercises was always very small suggesting that the work was done in a consistent manner. However, large errors were often detected between samples and bins. It is not possible to compare directly these errors with the standard deviations for the devices as these latter carry the errors associated with the grain. However, the general indication is that they may contribute to much of the variation within a device.

With specific quality parameters, the calculated errors shown in Table 7 and also illustrated in Figs 5 -7, indicate that the largest variation occurred in measuring total impurities, broken grains, and 100-grain weight. Errors in the region of 15 - 25% for an assessment based on a single sample were apparent. Errors for moisture content and specific weight were significant but much smaller. However, even with moisture content, the range of values between the highest and lowest samples was 1.1% of moisture. It must be assumed that all these errors are a result of a stratified distribution, particularly of fine material and broken grains, created as the grain was loaded in a bin, thus resulting in a clumped distribution. These results are exactly in line with those obtained for fine material in the Detection of Insects experiment reported above. However, some caution should be used when considering the data in the Figures as they represent values created by a statistical process rather than direct experimentation. Therefore, they should be used as guidance rather than an exact representation of the effects of changing sampling frequency.

Practical implications

The practical implications of these data are that rejection of a load of grain for excess fine material, broken grains or for 100-grain weight, on the basis of results from a single sample, may be difficult to justify. However, the data in Figs 5 - 7 show that the error can be reduced to some extent by taking more samples. Even greater reductions in the errors of prediction of the quality of a large bulk of grain are possible if the results of analysis from each of the individual lots making up the bulk, are pooled. This could be of particular significance when monitoring input quality for a production process or when filling a large ship or store. However, the results of this investigation suggest that it may never be possible to estimate the total impurities, broken grain or 100-grain weight to within an accuracy of better than 5 - 10%.

3. General conclusions and recommendations

This investigation would appear to be the first of its kind in the U.K. and, perhaps, in the world. Therefore, it is difficult to draw comparisons with or support from other work and some caution is warranted when considering the implications of the results and in using them to recommend changes in current practice. However, the work was extensive and parts of it were confirmed by scientists in France, so it is reasonable to draw some conclusions and offer some suggestions for further development.

As stated in Section 1, there is considerable variation in the way in which grain is sampled in the U.K. and there is a lack of documented instruction on sampling methods or the use of sampling equipment. This would appear to be unsatisfactory, particularly if the implementation of BS 5750 is to be taken seriously. It would appear that the equipment used to collect the sample has little effect on the results obtained. However, this conclusion may only apply when the equipment is used in accordance with Standard Operating Procedures by fully trained staff.

This work has yielded a large body of evidence to support the conclusion that the detection of low (below five insects/kg) populations of insects by the collection and examination of samples is extremely unreliable. Therefore, when taking grain into store there must be a very high chance that in some cases insects will be missed. The old rule that "not finding insects does not confirm the grain is uninfested" is still correct and emphasises the need for taking appropriate precautions at the start of and during storage. The difficulties in detection may be less serious from a commercial standpoint when dealing with traded grain. Provided the supplier's method of pest detection is as good or better than that used by the customer, it is unlikely that insects not detected by trapping during storage or by sampling during out-loading will be found on receipt.

The author is not aware of data to indicate that low numbers of insects in grain represent any risk to consumers. However, there is some circumstantial evidence to suggest that insects or insect fragments in food may pose a risk to health. Clear evidence connecting insects with, for example, food allergies, would necessitate a change in attitude to the detection of low numbers of insects. Currently, the only option for improving rapid detection of insects would appear to be to increase the size of sample examined. Improved methods of assessing 1kg samples will do nothing to increase detection of low population densities in lorry-loads of grain unless they are sufficiently rapid to allow many kg to be examined in one or two minutes. Perhaps the most obvious idea worthy of investigation and development, would be a self-feeding, automatic sieve that would process rapidly 20kg of grain and separate out any insects.

Very little indication of bias in the results caused by the device used to collect samples, was detected. A reasonable suggestion is that, although there may be variation between devices, this is masked by the variation of the grain between samples and between bins. However, much more work is needed to provide precise data on the exact contribution to the total error made by individual variables.

This investigation suggests that it is difficult to estimate some common quality parameters with any degree of accuracy, as grain is delivered to or discharged from a store in lorry-loads. In particular, the estimation of total impurities, broken grains and 100-grain weight would appear to carry the risk of sufficiently large errors to make it inadvisable to reject loads of grain on the basis of results from a single sample unless the grain was outside specification by 20% or more. Fortunately, characteristics such as moisture content and specific weight can be estimated with more precision. However, even here the single samples gave greater errors than were obtained if multiple assessments were made. The practice of rejecting a load on the basis of the detection of a single insect is, of course, fully justified, as the one insect found represents only a very small part of the likely population in the load.

It is very encouraging to observe that the accuracy of prediction of all quality parameters shows a marked improvement if the results from individual loads are pooled. In this way, moving averages can be constructed that will give the best practical assessment of the quality of incoming or outgoing grain, even though the predictions of the quality of individual loads may carry errors of up to 25%.

This latter observation must cast some doubt on current intake monitoring practices, in which individual loads are classed as acceptable or unacceptable. Rejection is always costly to the supplier and can be expensive to the intended recipient, who may have to find alternative grain at short notice. It may be worth considering the idea of contracts that are based on a quality band, with bonuses and penalties as appropriate. The final assessment of quality would be retrospective and determined on the basis of pooled quality assessments from individual loads. This would go some way towards overcoming problems caused by rejection and the inherent variable nature of grain. It would appear that this approach is being followed already to a lesser or greater degree by a few important users of grain in the U.K., Europe and the U.S.A. In some instances, all responsibility for monitoring and maintaining quality is placed on the supplier and no intake sampling is carried out by the customer. This process is reputed to yield dramatic costs savings for the customer and routine audits are used to confirm that the suppliers stick to the specified quality standards.

Obviously, this process can not be applied to the detection of live pests in export grain, to meet phytosanitary requirements, using existing procedures. The regulations state that "the grain must be examined by an appropriate method and be found free from quarantine pests and practically free from other injurious insects". However, modern methods of detection and prevention of infestation during storage can be so effective that suppliers of grain who have not detected insects by trapping, or who have followed an appropriate storage strategy, can have almost total confidence that no insects will be found in their grain as it is loaded onto ships.

Given more research, particularly on sampling and sampling statistics, the above procedures may have much wider implications for the U.K. grain industry. There would appear to be many flaws in attempting to establish the quality of grain at the time of intake into a store, ship or processor. However, quality control must be applied in all these cases. It may be worth considering a process of quality assurance based on assessments performed by the supplier of the grain rather than the receiver. This will give the possibility of very much better detection of pests, as well as reasonably accurate estimates of the overall quality of a bulk of grain. Buyers could make their selection of grain based on data collected and supplied by the seller and apply occasional technical audits to confirm that quality parameters were being met. Such an approach has been adopted by individual companies throughout the world and is reputed to have produced benefits for both suppliers and users.

Arising from this investigation a number of recommendations can be made:-

i) This investigation represents only the preliminary steps into a full investigation of grain sampling and the development of a modern process control approach. Such an approach has the potential to save money for producers and users of grain, and to lead to an improvement in the quality and consistency of U.K. grain. Therefore, further studies on this topic should be undertaken.

ii) Standard Operating Procedures should be produced for methods of grain sampling and use of samplers. Specifications for grain sampling devices should also be produced. These should be used to up-date the current BS Standard on grain sampling.

iii) Some R&D should be set up to develop an automated grain sieve capable of removing insects from large samples of grain.

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Appendix 1 - use of sampling equipment

1. Gravity spear

   i) Ensure that the sample cup is clean and completely empty.

   ii) Attach rods as appropriate.

   iii) Insert the spear, if possible in a single movement. Do not pull the spear upwards until the required depth has been reached. Take particular care not to open the sample cup when attaching or detaching rods as the spear is inserted

   iv) At the required depth, pull the spear rod up about 2.5cm to open the sample cup and hold in that position for 20 seconds.

   v) Withdraw the spear and remove the sample, ensuring that the cup is fully emptied.

   Note: if the sample cup is not full when the spear is withdrawn from the grain, the sample must be discarded and another sample collected.

2. Compartmented spear

   i) Ensure that all the compartments are clean and empty.

   ii) Ensure that the spear is in the "closed" position.

   iii) Insert the spear fully into the grain.

   iv) Open the spear fully and leave for 20 seconds.

   v) Close the spear.

   vi) Withdraw the spear and empty out the grain into a suitable length of plastic gutter. Ensure that all the material is extracted from every compartment.

   Note: if any of the compartments are not full when the spear is withdrawn, that sample should be discarded and the operation repeated.

3. Vacuum sampler

   i) Ensure that the sample container is empty and clean. Check that the filter is free from dust and the hose and pipes are clean and empty.

ii) Connect pipes as appropriate.

iii) Whilst holding the intake of the sample pipe clear of the surface of the grain, turn on the motor.

iv) Allow the intake to rest on the grain and descend under its own weight. If necessary apply slight pressure. As the sample tube descends into the grain, apply downward pressure as necessary to maintain a constant rate of descent.

v) When the sample tube reaches the bottom of the bin, or the required depth, turn off the motor. The grain in the sample container will be approximately equivalent to the volume of the sample tube and can be considered as a core sample from the top to bottom of the bin.

vi) When emptying the sample container make sure that any dust on the filter is shaken off into the grain and thoroughly mixed in.

Note: if a sample is required from a specific depth, the sample tube should be sucked down to the desired depth, the motor stopped and the container emptied. The motor should then be restarted and a sample collected.

It is extremely difficult to regulate the amount of grain collected by vacuum samplers. The sample size is affected by the length of pipe inserted into the grain and its rate of insertion.

Appendix 2 - Parameters for quality determination

Weigh total sample of grain.

Measure specific weight of whole sample using chondrometer.

Obtain a sample of 200-250g by use of a divider. Pass through 2.00mm and 0.71mm mesh sieves (30 shakes). Examine material retained on 0.71mm sieve, and that passing through, for insects.

A. Obtain another sample of about 250g, by use of divider. Weigh. Sieve, using 3.5mm and 1.0mm slotted sieves, for 30 seconds on a sieving machine. Examine the material retained on the 3.5mm sieve. Return large grains of the basic cereal, grains still in husk and grains of other cereals to the sifted sample. Combine the material left on the 3.5mm sieve with that passing through the 1.0mm sieve. Weigh and enter the result at B.

B. Impurities remaining from A (g)

C. B expressed as a percentage $\frac{B \times 100}{A}$ (%)

D. Divide the sifted sample on sample divider to obtain a partial sample of 50-70g. Weigh. Spread this sample out. De-husk any grains still in husk.

E. Corrected weight of partial sample $\frac{D \times 100}{100-C}$ (g)

F. Separate from the partial sample the following classes and sub classes of impurities.

Record weights and calculate percentages by:- $\frac{\text{weight} \times 100}{E}$

G. **Broken grains** (%)

H. **Sprouted grains** (%)

I. **Miscellaneous impurities:-**

i) Noxious seeds (%)

ii) Ergot (%)

iii) Weed seeds, damaged grains, extraneous matter, husks, decayed grains, dead insects, insect fragments, grains of other cereals which are damaged, decayed or affected by ergot. (%)

J. Add together percentages from C + Ii) + Iii) + Iiii) to give the total percentage of miscellaneous impurities.

K. **Grain impurities:-**

i) Other cereals and pest damaged grains

ii) Grains with discoloured germs (not applicable to barley). Enter the percentage in excess of 8%

iii) Add together percentages i) and ii)

iv) Grains damaged by heat during drying

v) Frost-damaged and unripe (green) grains. Separate these then combine with shrivelled grains.

vi) Shrivelled grains. After removing all the above types of impurities, sieve the sample for 30 seconds using sieving machine, using 2.0mm sieve for wheat and 2.2mm sieve for barley.

Combine weights of v) and vi).

L. Add together percentages from Kiii) + Kiv) + Kvi) to give the total percentage of grain impurities.

M. Add together percentages from G + H + J + L to give the **Total Percentage of Impurities.**

Count out 100 grains from the final sieved sample of "perfect" grains. Weigh to give the 100 grain weight.

Carry out moisture content determinations on two samples simultaneously of the final sieved sample using the oven method - BS4317 : Part 3 : 1987.

Table 1. Mean numbers of dead insects and dyed grains recovered from grain artificially contaminated with dead, adult *Oryzaephilus surinamensis, Sitophilus granarius* and dyed grains. The mean sample weight and mean weight of fine material/sample are also given. The figures in parentheses are standard deviations.

	Sample wt. g	Fines g	Insects/sample O. sur.	S. gran.	Total	Dyed grains /sample
Inclusion rate of 1/5kg						
Gravity spear	208 (8.5)	0.4 (0.2)	0.03 (0.2)	0	0.03 (0.2)	0
Compartmented spear	259 (20.6)	0.3 (0.2)	0.04 (0.2)	0	0.04 (0.19)	0.04 (0.19)
Inclusion rate of 1/kg						
Gravity spear	223 (18.9)	1.5 (0.3)	0.1 (0.3)	0.1 (0.3)	0.2 (0.3)	0.03 (0.6)
Compartmented spear	273 (28.2)	1.2 (0.2)	0.2 (0.4)	0.2 (0.4)	0.3 (0.4)	0.1 (0.5)
Inclusion rate of 5/kg						
Gravity spear	239 (23.5)	1.0 (0.4)	0.1 (0.5)	0.3 (0.3)	0.4 (0.5)	0.7 (2.1)
Compartmented spear	285 (14.5)	1.2 (0.7)	0	0.4 (0.8)	0.4 (0.8)	0.8 (0.9)

Table 2. The numbers of dead insects and dyed grains found per kg in samples removed from wheat to which dead, adult insects and dyed grains had been added at three inclusion rates.

	Inclusion rate/kg	Insects/kg	Dyed grains/kg
<u>Compartmented spear</u>			
	0.2	0.1	0.1
	1.0	1.5	0.9
	5.0	6.0	3.2
<u>Gravity spear</u>			
	0.2	0.1	0.1
	1.0	1.5	0.9
	5.0	5.8	3.0

Table 3. The number of grain samples containing one or more dead insects or dyed grains.

	Inclusion rate/kg	No. samples with 1 or more insects	No. samples with 1 or more dyed grains
<u>Gravity spear</u> (81 samples)			
	0.2	1	0
	1.0	21	6
	5.0	33	44
<u>Compartmented spear</u> (27 samples)			
	0.2	1	1
	1.0	8	3
	5.0	15	15

Table 4. Means of the three replicates each taken at three sampling points with each device when sampling 20-tonne bulks of wheat.

Exercise	Bin/ point	Sample device	Specific wgt (kg/hl)	Total impurities (%)	Moisture content (%)	Broken grains (%)	100 grain wgt (g)
1	1	gravity	73.3 (0.17)	5.95 (0.92)	14.4 (0.07)	3.63 (0.64)	- -
	1	vacuum	- -	5.97 (0.51)	14.5 (0.06)	3.29 (0.39)	- -
	1	compart	73.6 (0.09)	5.25 (0.01)	14.4 (0.05)	3.21 (0.06)	- -
	2	gravity	75.3 (0.36)	5.82 (0.67)	14.3 (0.05)	3.22 (0.51)	- -
	2	vacuum	- -	5.79 (0.43)	14.6 (0.20)	3.03 (0.18)	- -
	2	compart	76.1 (0.36)	7.59 (1.27)	14.3 (0.06)	3.39 (0.47)	- -
	3	gravity	75.5 (0.33)	8.72 (1.36)	14.6 (0.15)	3.24 (0.34)	- -
	3	vacuum	- -	8.96 (0.14)	14.6 (0.08)	3.29 (0.23)	- -
	3	compart	76.1 (0.31)	9.38 (0.59)	14.6 (0.09)	3.58 (0.53)	- -
2	1	gravity	72.8 (0.73)	4.99 (0.57)	14.5 (0.08)	2.56 (0.36)	- -
	1	vacuum	- -	5.93 (0.52)	14.3 (0.17)	3.27 (0.38)	- -
	1	compart	74.3 (0.08)	5.60 (0.78)	14.3 (0.01)	3.07 (0.45)	- -
	2	gravity	75.4 (0.33)	5.82 (0.04)	14.2 (0.10)	3.55 (0.12)	- -
	2	vacuum	76.3 (0.14)	6.30 (0.69)	14.3 (0.08)	3.65 (0.37)	- -
	2	compart	76.8 (0.31)	5.37 (0.28)	14.2 (0.05)	3.08 (0.14)	- -
	3	gravity	74.3 (1.58)	5.66 (0.69)	14.5 (0.12)	2.95 (0.41)	- -
	3	vacuum	75.6 (0.37)	7.02 (0.96)	14.6 (0.05)	2.73 (0.18)	- -

	3	compart	76.3 (0.39)	8.12 (0.48)	14.5 (0.08)	3.42 (0.11)	- -
3	1	gravity	74.3 (0.34)	5.81 (0.58)	13.5 (0.91)	3.11 (0.28)	3.84 (0.06)
	1	vacuum	75.4 (0.62)	5.84 (1.51)	14.1 (0.13)	3.16 (0.86)	3.61 (0.16)
	1	compart	76.5 (0.37)	5.74 (0.26)	14.0 (0.06)	3.02 (0.07)	3.83 (0.08)
	2	gravity	72.7 (0.34)	5.34 (0.47)	14.0 (0.11)	2.59 (0.56)	3.24 (0.10)
	2	vacuum	73.5 (0.36)	4.48 (0.05)	14.1 (0.16)	2.22 (0.13)	3.30 (0.20)
	2	compart	74.4 (0.12)	5.01 (0.45)	13.9 (0.09)	2.40 (0.26)	3.24 (0.05)
	3	gravity	74.6 (0.37)	6.38 (0.36)	14.1 (0.08)	3.08 (0.14)	4.04 (0.24)
	3	vacuum	75.7 (0.38)	6.30 (0.44)	14.2 (0.14)	3.03 (0.23)	3.88 (0.08)
	3	compart	76.2 (0.14)	5.50 (0.21)	14.2 (0.01)	2.59 (0.21)	4.22 (0.29)

Figures in parenthesis are the standard deviations about the mean values.
- denotes where there was an insufficient volume of grain to take a measurement.

Table 5. Means of the three replicates taken at three sampling points with each device when sampling malting barley under field conditions.

Point	Sample device	Specific weight (kg/hl)	Total impurities (%)	Broken grains (%)
1	gravity	72.4	2.64	0.58
		(0.26)	(0.18)	(0.03)
1	vacuum	73.6	2.40	0.67
		(0.05)	(0.11)	(0.08)
1	compart	72.9	2.63	0.59
		(0.40)	(0.31)	(0.09)
2	gravity	72.2	3.15	0.86
		(0.46)	(0.26)	(0.07)
2	vacuum	72.2	5.04	0.88
		(0.17)	(0.17)	(0.16)
2	compart	72.7	2.98	0.68
		(0.22)	(0.03)	(0.01)
3	gravity	72.5	2.98	0.79
		(0.09)	(0.45)	(0.31)
3	vacuum	72.9	4.63	1.31
		(0.21)	(0.27)	(0.05)
3	compart	-	4.32	1.01
		-	(0.34)	(0.16)

Figures in parenthesis are the standard deviations about the mean values.
- denotes where there was an insufficient volume of grain to take a measurement.
NB. moisture content determination of the samples was omitted on this occasion.

Table 6. The mean results obtained with, and the variation attributed to, three different sampling devices when collecting samples from wheat or barley.

Commodity	Sample device	Specific wgt (kg/hl)	Total impurities (%)	Moisture content (%)	Broken grains (%)	100 grain weight (g)
Wheat (n=27)	gravity	74.2 (1.2)	6.1 (1.2)	14.2 (0.5)	3.1 (0.5)	3.7### (0.4)###
	vacuum	75.1# (1.2)#	6.2 (1.4)	14.4 (0.2)	3.0 (0.5)	3.6### (0.3)###
	compart	75.6 (1.1)	6.4 (1.6)	14.3 (0.2)	3.1 (0.5)	3.8### (0.4)###
Barley (n=9)	gravity	72.4 (0.3)	2.9 (0.4)	-	0.7 (0.2)	-
	vacuum	72.9 (0.6)	4.0 (1.2)	-	1.0 (0.3)	-
	compart	72.6## (0.4)##	3.3 (0.8)	-	0.8 (0.2)	-

Figures in parenthesis are the standard deviations about the mean values.

- assessments not carried out.
21 values only due to insufficient grain in 6 of the samples.
8 values only due to insufficient grain in 1 of the samples.
9 values only.

Table 7. Typical sizes of error in assessment of quality standards of wheat or barley, attributable to differences between samples, sample position, between bins or between exercises. The errors are expressed in appropriate units for the quality parameter in question.

Quality parameter	Commodity	Source of error	Typical size of error
Specific weight (g/hltr)	Wheat	Between samples	1.07
		Between bins	0.67
		Between positions	--
		Between exercises	--
	Barley	Between samples	0.44
		Between positions	0.25
		Between lots	--
		Between exercises	na
Total impurities (g)	Wheat	Between samples	0.90
		Between positions	0.12
		Between bins	1.12
		Between exercises	0.32
Broken grains (%)	Wheat	Between samples	0.45
		Between bins	0.23
		Between positions	--
		Between exercises	0.21
	Barley	Between samples	0.18
		Between positions	0.20
		Between lots	0.20
		Between exercises	na
Moisture content (%)	Wheat	Between samples	0.24
		Between bins	0.13
		Between exercises	0.23
		Between positions	--
100-grain weight (g)	Wheat	Between samples	0.19
		Between positions	0.08
		Between bins	0.39
		Between exercises	na

na signifies that there were insufficient data for the analysis
-- signifies that the error was insignificant

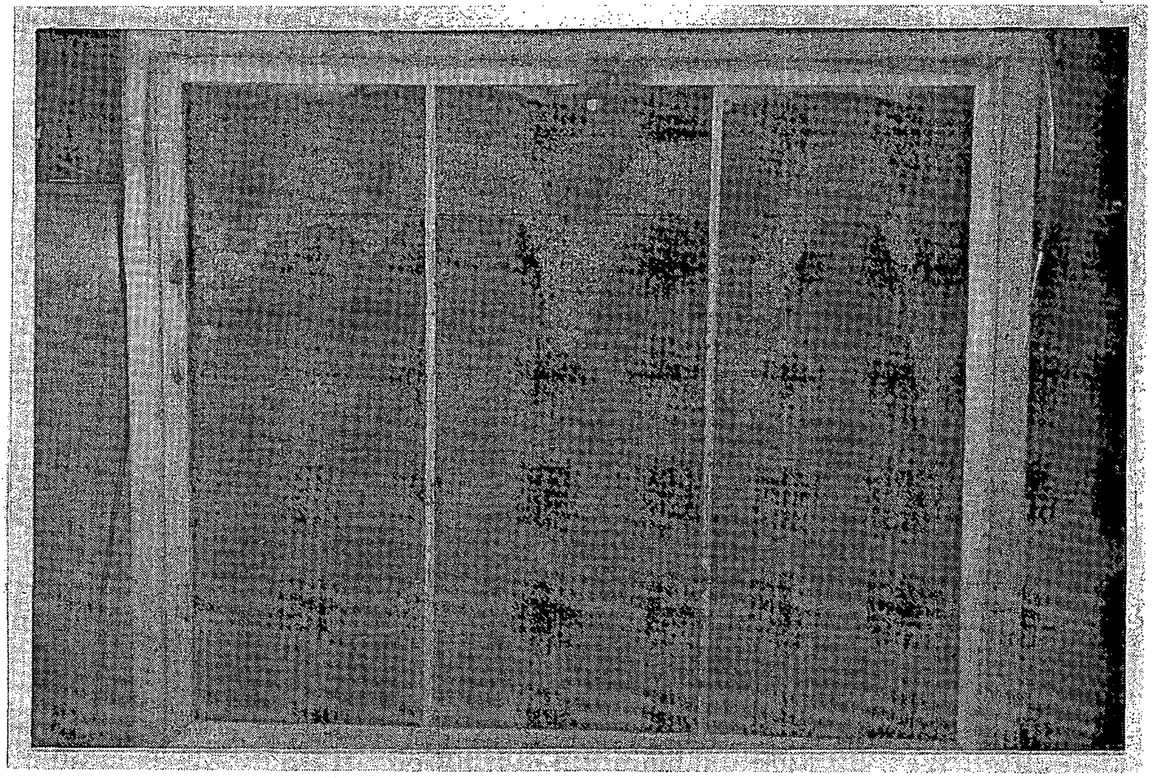

Figure 1. Disturbance of grain caused by insertion of a spear.

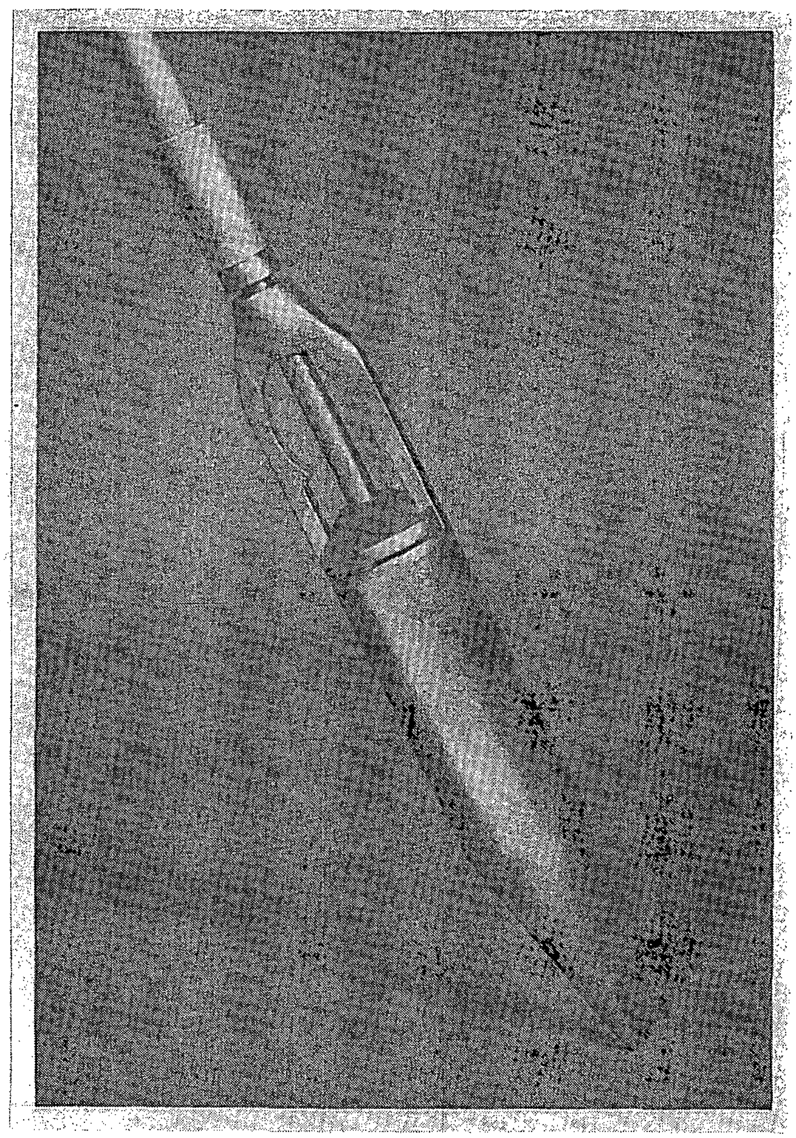

Figure 2a. Gravity spear.

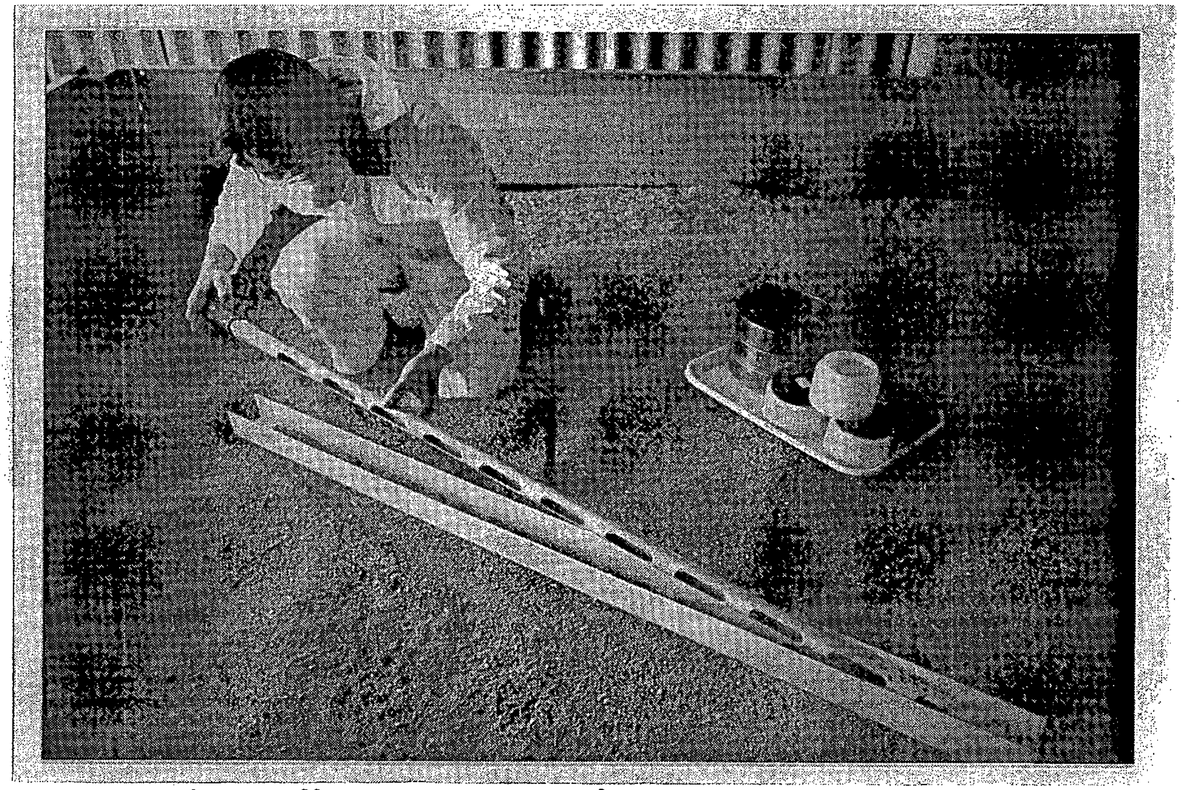

Figure 2b. Compartmented spear.

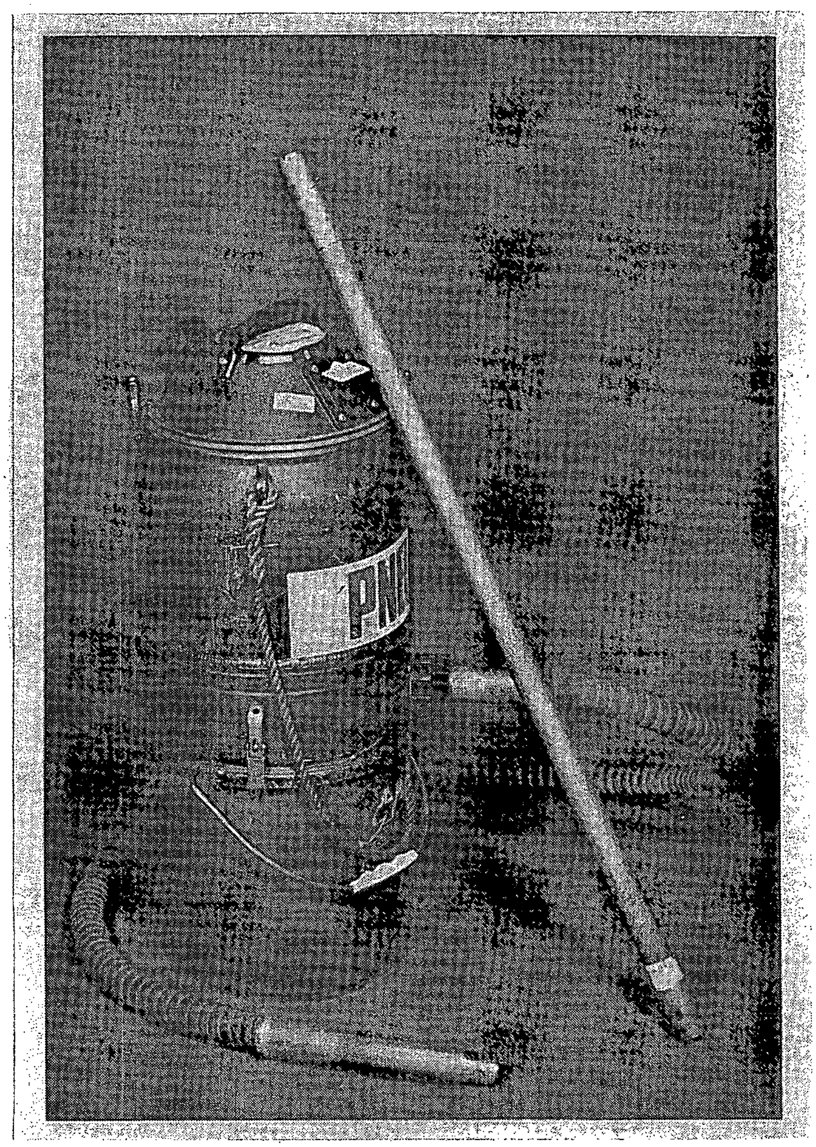

Figure 2c. Vacuum sampler.

Fig. 3 Predicted errors when sampling 20 tonne lots of wheat for Specific Weight. Each sample was taken from a different position in a lot with any device, and an overall mean calculated.

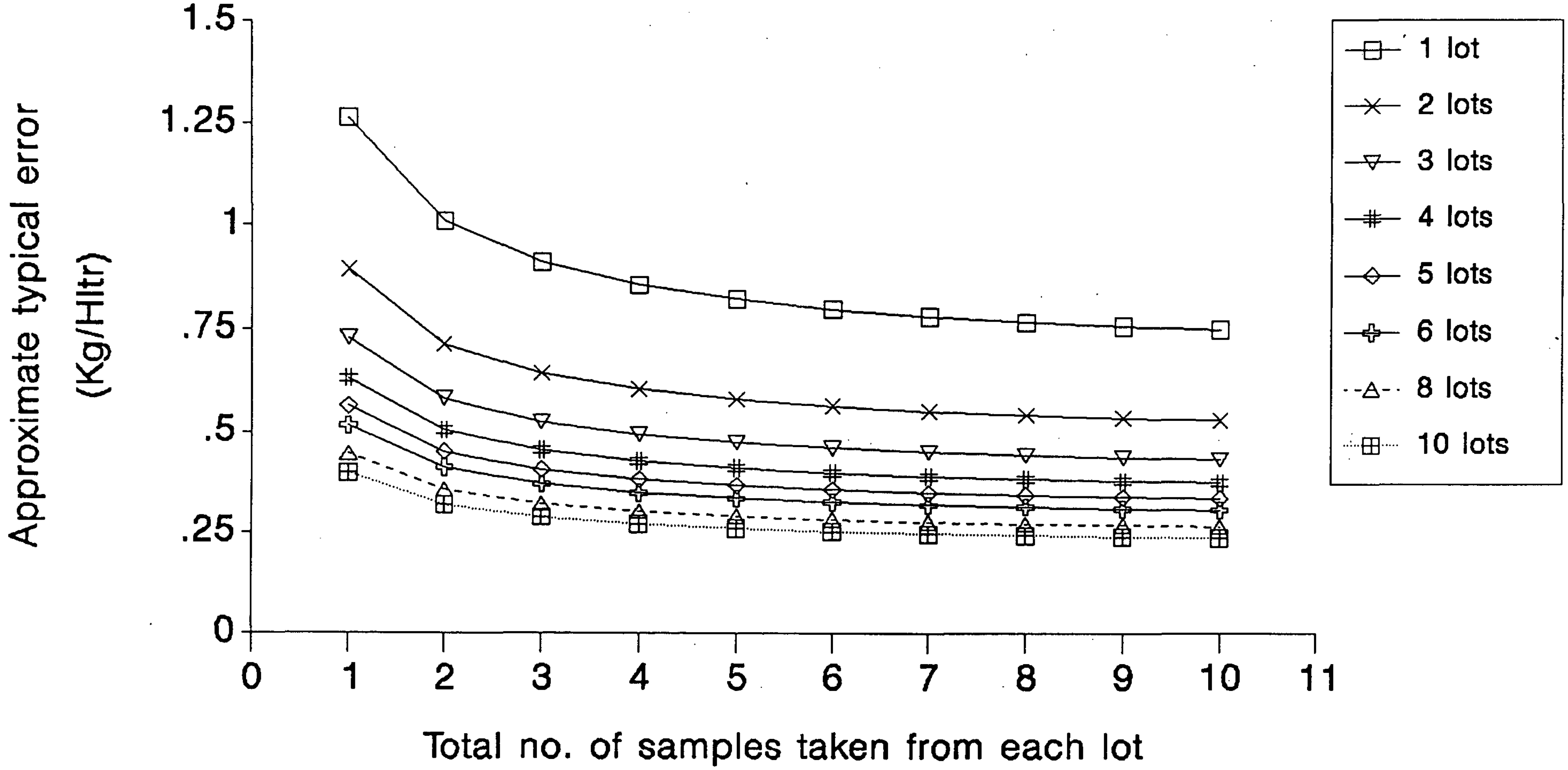

Fig. 4 Predicted errors when sampling 20 tonne lots of wheat for Total Impurities. Each sample was taken from a different position in a lot with any device, and an overall mean calculated.

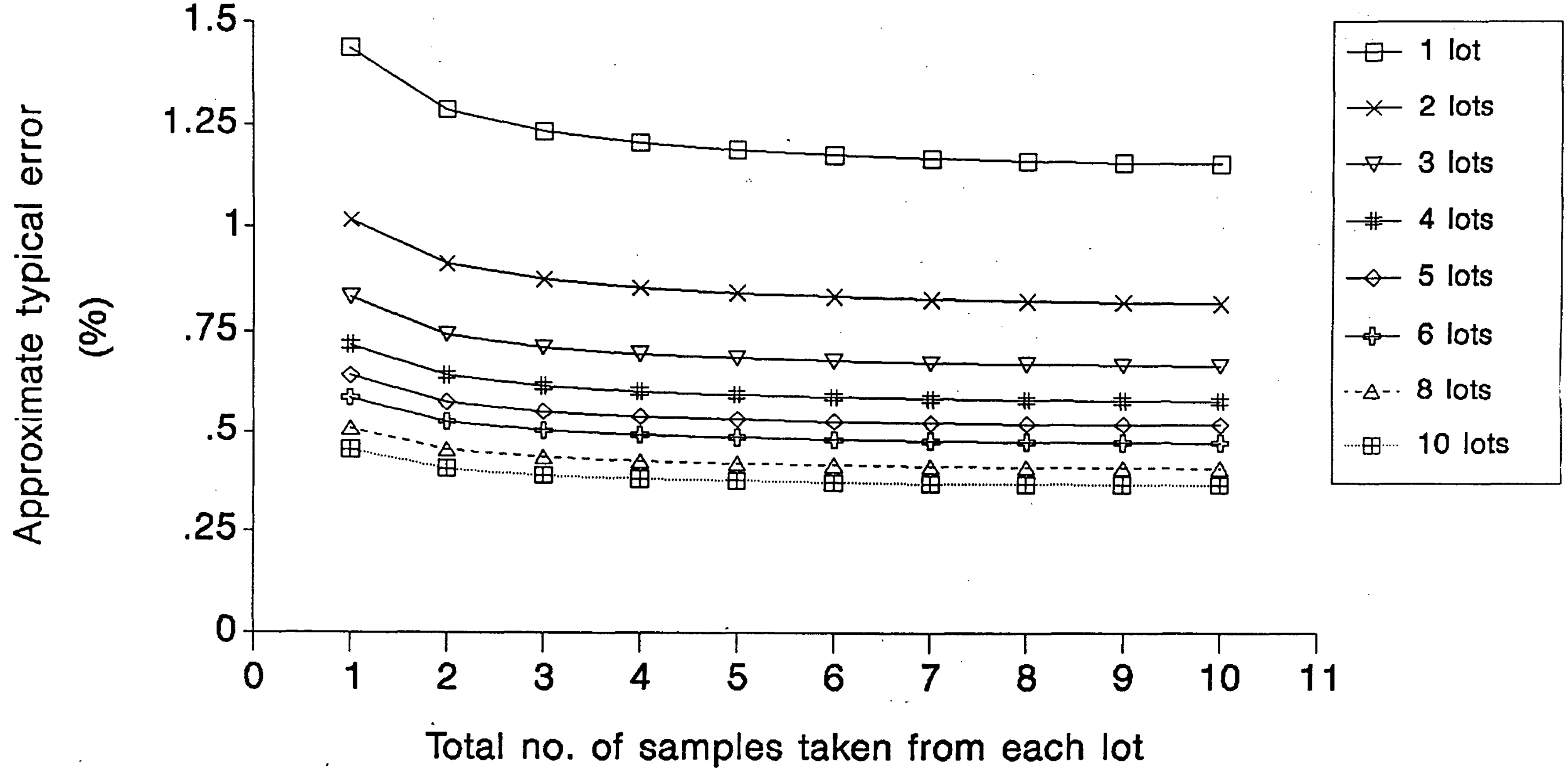

Fig. 5 Predicted errors when sampling 20 tonne lots of wheat for Moisture Content. Each sample was taken from a different position in a lot with any device, and an overall mean calculated.

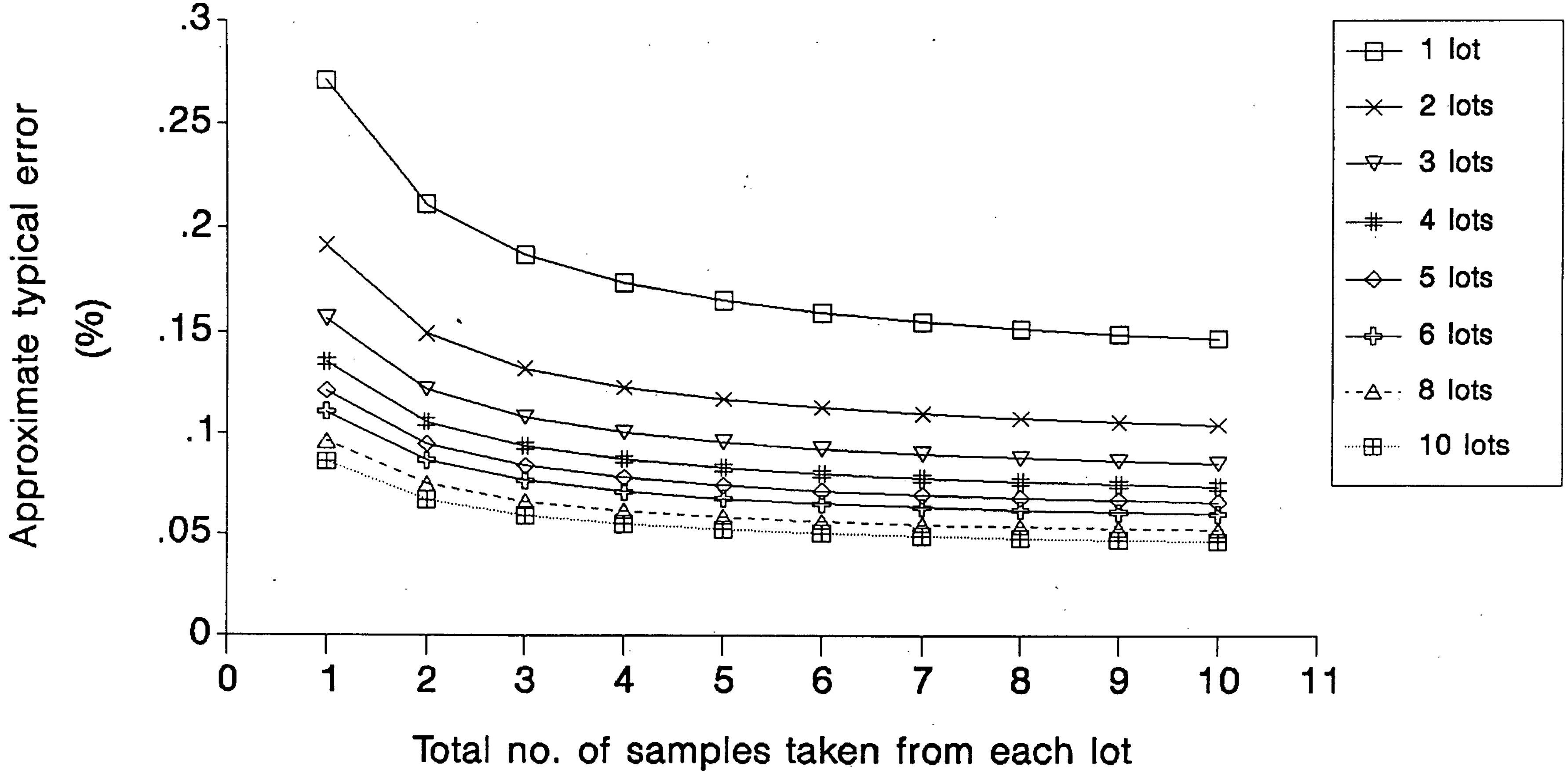

Fig. 6 Predicted errors when sampling 20 tonne lots of wheat for Broken Grains. Each sample was taken from a different position in a lot with any device, and an overall mean calculated.

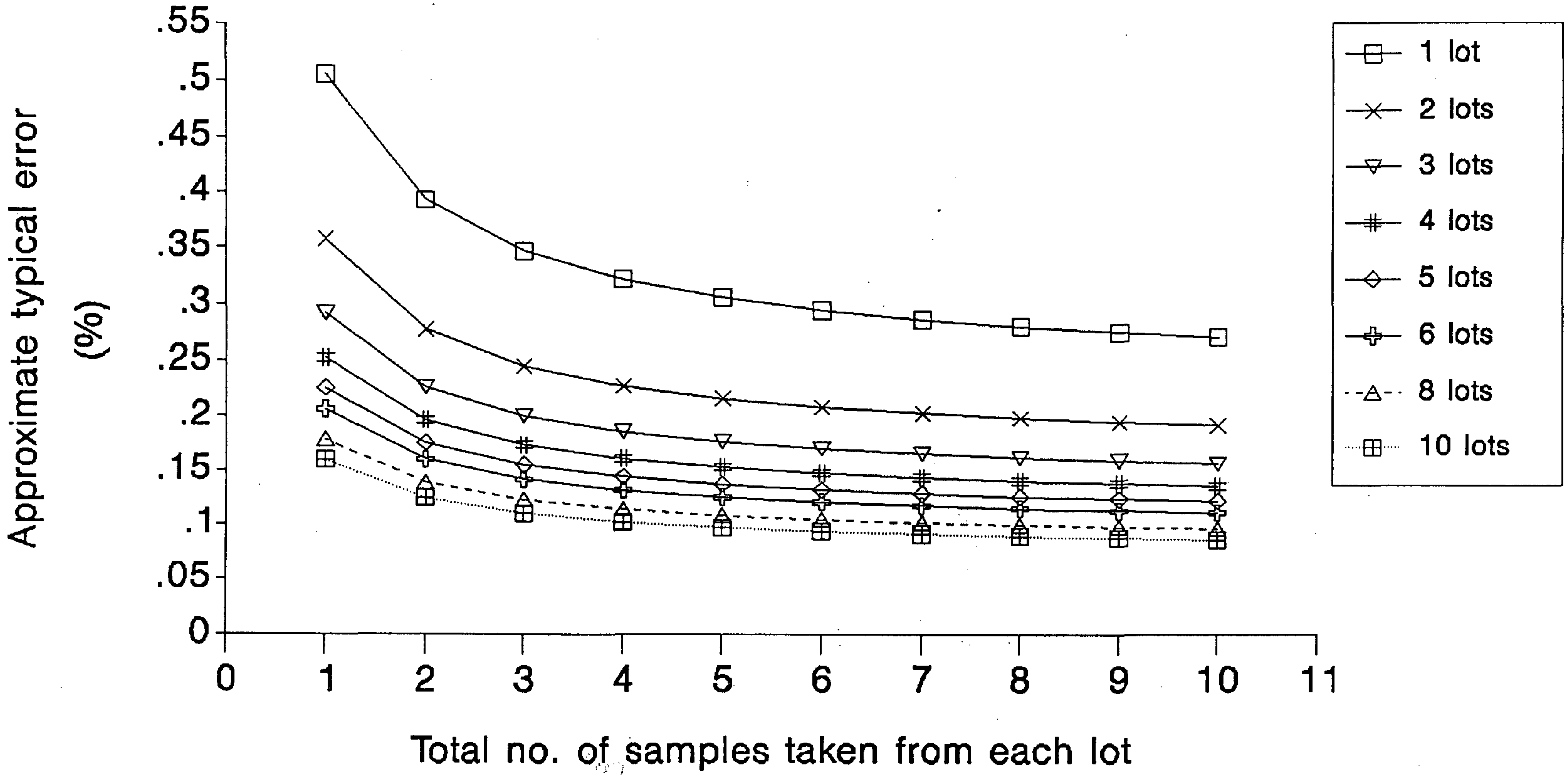

Fig. 7 Predicted errors when sampling 20 tonne lots of wheat for 100-Grain Weight. Each sample was taken from a different position in a lot with any device, and an overall mean calculated.