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Foodstuffs — Methods of analysis for the detection of genetically modified organisms and derived products — Sampling

Produits alimentaires — Méthodes d'analyse pour la détection des organismes génétiquement modifiés et des produits dérivés — Échantillonnage

ICS 67.050

ISO/CEN PARALLEL ENQUIRY

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 21568 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*, Subcommittee SC , and by Technical Committee CEN/TC 275, *Food analysis - Horizontal methods* in collaboration.

Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

Since the first draft prEN ISO 21568 has been circulated for the parallel enquiry in 2003, new scientific approaches to sampling have been elaborated. The results of a large study on distribution of GMOs in loose material (bulk agricultural commodities) will be submitted for publication within the next months. Additionally, a sampling protocol based on these findings was published as a Recommendation by the European Commission in November 2004. In this study it was proved clearly, that different lots of comparable size showed a large variety of GMO distribution which is of global relevance. This leads to the conclusion that a protocol which is based on a distribution assumption can only yield erroneous results. Therefore, it was necessary to take these new developments into account and to publish a second draft European International Standard.

Based on the new scientific evidence the chapter 6 "Sampling of loose food products" of prEN ISO 21568:2003 was changed as follows:

A new statistical approach was introduced. While the old version suggested a sampling protocol which was based on a distribution assumption and therefore was based mainly on the size of the laboratory sample to be analysed, the new protocol is independent of the distribution of GMOs in the lot, and is therefore not affected by stratification or clustering effects within the lot. In addition to that the new protocol allows an estimation of the sampling error, which is of great benefit for enforcement authorities as well as for trading companies.

All other changes to the prEN ISO 21568:2003 were introduced following the comments received during the first enquiry.

Introduction

Correct sampling is an operation that requires the most careful attention. Emphasis should be laid on the necessity of obtaining a representative sample of the goods under investigation. Accurate analytical work and interpretation of results are wasted if the sample does not accurately represent the lot from which it is taken.

If ad-hoc sampling of food products is undertaken without applying a sampling strategy and without considering the lot specific properties, the analytical result is only valid for the sample which has been analysed. It is not possible to extend the result to the rest of the lot.

By applying sampling frameworks to assess the level of compliance of a given lot of products a certain number of samples has to be taken, and the result of the analysis can be extended to the whole lot. The use of sampling plans is the only effective way to make correct statements about the nature, in this case the GMO-content, of the product tested.

The procedures given in this standard are recognised as good practice and it is strongly recommended that they be followed whenever practicable. It is recognised that it is difficult to lay down fixed rules to be followed in every case, and particular circumstances may render some modification of the method desirable.

This standard has been established for food products, but could also be applied to other products, e. g. feed and environmental samples.

NOTE In certain areas there are widely recognised trade associations which specify rules for the sampling plans to be used in contracts under their auspices. In no case will this standard override the rules laid down in such contracts.

1 Scope

This Standard gives guidance for setting up valid sampling strategies for food products that are to be analysed for the presence of genetically modified organisms and derived products.

2 Normative reference

This draft standard incorporates, by dated or undated reference, provisions from other publications. The normative references are cited at the appropriate places in the text, and the publications re-listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this draft standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

ISO 13690, Cereals, pulses milled products — Sampling of static batches.

ISO 6644, Flowing cereals and milled products – Automatic sampling by mechanical means

EN ISO 21572:2004, Foodstuffs - Methods of analysis for the detection of genetically modified organisms and derived products - Protein based methods

ISO/FDIS 21569, Foodstuffs - Methods of analysis for the detection of genetically modified organisms and derived products - Qualitative nucleic acid based methods

EN ISO 21571:2005, Foodstuffs - Methods of analysis for the detection of genetically modified organisms and derived products - DNA extraction methods.

ISO/DIS 24276, Foodstuffs - Nucleic acid based methods of analysis for genetically modified organisms and derived products - General guidelines and requirements.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
Consignment
A quantity of some commodity delivered at one time and covered by one set of documents. The consignment may consist of one or more lots or part of lots.

[ISO 7002:1986]

3.2
Lot
stated portion of the consignment to be tested for presence of GMO.

3.3
Increment
a quantity of material taken at one time from a larger body of material.

NOTE Increments may be tested individually aiming at estimation of the variation of any characteristic throughout a lot (or between lots).

[ISO 7002:1986]

3.4
Item
an actual or conventional object (a defined quantity) on which a set of observations may be made.

[ISO 7002:1986]

3.5
Sample
one or more items (or a portion of material) selected in some manner from a lot. It is intended to provide information representative of the lot, and, possibly, to serve as a basis for decision on the lot.

3.6
File increment sample
an increment that is retained for a specific period of time for further analysis

3.7
Bulk sample
a composite of the increments taken from a lot.

3.8
Laboratory sample
a sample as prepared for sending to the laboratory and intended for inspection or testing

[ISO 7002:1986]

3.9
Test portion
a sample, as prepared for testing or analysis, the whole quantity being used for analysis or testing at one time

[ISO 3534-1]

3.10
Lot size
the number of items or quantity of material constituting the lot

[ISO 7002:1986]

3.11
Sample size
the number of items or quantity of material constituting the sample

[ISO 7002:1986]

3.12
Sample division
the process of selecting one or more representative subsamples from a sample by such means as riffling or mechanical dividing

3.13
Estimation error
in the estimation of a parameter, the estimation error is the difference between the calculated value of the estimator and the true value of this parameter

3.14
Sampling error
part of the total estimation error due to one or several of the following parameters:

the failing to accurately represent the lot;

the random nature of sampling;

the known and accepted characteristics of the sampling plans.

3.15
Sampling plan
the predetermined procedure for the selection, withdrawal and preparation of samples from a lot to yield the required information so that a decision can be made regarding the acceptance of the lot.

[ISO 7002:1986]

3.16
Acceptance quality limit (AQL)
quality level that is the worst tolerable process average when a continuing series of lots is submitted for acceptance sampling

NOTE This concept only applies when a sampling scheme with rules for switching and for discontinuation, such as in ISO 2859 or ISO 3951, is used.

NOTE Although individual lots with quality as bad as the acceptance quality limit may be accepted with fairly high probability, the designation of an acceptance quality limit does not suggest that this is a desirable quality level. Sampling schemes found in International Standards such as this part of ISO 2859, with their rules for switching and for discontinuation of sampling inspection, are designed to encourage suppliers to have process averages consistently better than the AQL. Otherwise, there is a high risk that the inspection severity will be switched to tightened inspection under which the criteria for lot acceptance become more demanding. Once on tightened inspection, unless action is taken to improve the process, it is very likely that the rule requiring discontinuation of sampling inspection pending such improvement will be invoked.

[ISO 2859-1]

3.17
Inspection level
the inspection level relates the sample size to the lot size and hence to the discrimination afforded between "good" and "poor" quality.

NOTE A sampling scheme involves "switching" between normal, tightened and reduced inspection sampling plans, (see e.g. Table 1 of ISO 2859-1:1989 and Table 1-A of ISO 3951:1989).

3.18
Specification limit
a limit (value) set by a contract or legal requirements above which a decision shall be made, e.g. regulatory compliance

3.19
Non conforming item or increment
a sample from which a test result above the specification limit has been obtained.

4 Principle

In this standard sampling is considered to consist of the following steps:

— collection of a sufficient number of increments to form the bulk sample;

— reduction of the bulk sample to the laboratory sample;

— homogenisation and reduction of the particle size by appropriate means to form the analytical sample;

— determination of sampling error, if necessary.

Samples shall be representative of the lots from which they are taken. Therefore, as the composition of a lot is seldom uniform, a sufficient number of increments shall be taken and carefully mixed, thus giving a bulk sample from which the laboratory sample is obtained by successive divisions or otherwise.

If it is necessary to determine the sampling error, file increment samples should be kept for further analysis.

All sampling operations shall be carried out over a sufficiently short period of time so as to avoid any alteration in the composition of the samples.

5 Apparatus and equipment

Many different types of sampling instruments or equipment are available. Equipment should be chosen as appropriate for the food products to be sampled and the quantity and containers involved. Examples of sampling instruments are given in e. g. ISO 13690:1999, ISO 6644:2002. Special care is necessary to ensure that all sampling apparatus is clean to avoid contamination of the material under investigation.

Sampling shall be carried out in such a manner as to protect from adventitious contamination the samples, the sampling instruments and the container in which the samples are placed. Special attention shall be paid to avoid cross-contamination during the sampling procedure between different lots. Sampling apparatus shall be cleaned carefully, e. g. by using DNA-destroying agents. Material adhering to the outside of the sampling instrument shall be removed before the contents are discharged.

6 Sampling of non-packed food products

6.1 Statistical principles

Lot distribution properties affect the efficiency of sampling strategies [1]. In case the variable of interest is randomly distributed within the lot under investigation, the sampling error can be estimated according to the binomial distribution [2]. However, in reality, food product lots may show non-random distributions [3], and lot heterogeneity needs to be taken into account when defining sampling procedures statistically. This sampling

protocol also provides a way to estimate the sampling error associated with the overall GMO content of a lot, without imposing any distribution assumption. Indeed, estimates of sampling error as function of individual lot properties, are a pre-requisite to achieve a given risk level under any possible lot properties scenario.

The Standard Deviation (SD) of the increment GMO content estimates may be interpreted as an indicator of the lot heterogeneity and in highly heterogeneous lots an indication of the sampling error, generally the larger the standard deviation, the larger the sampling error in highly heterogeneous lots. When an estimation of the sampling error of the bulk sample is required, the users can execute the second step of the sampling protocol to determine the sampling error estimate.

When the analytical result obtained from the bulk sample is close to the specification limit and variability of the measurement is required, analysis of the individual file increment samples can be used to determine the error estimate related to this result.

NOTE: As an example for a specification limit = 3% and the acceptable range of the estimate is +/- 50%, it is recommended to proceed with the analysis of individual file increment samples if the estimate (x) falls within the range 1,5% < x < 4,5%.

6.2 Procedure

6.2.1 General

This protocol is based on a two step procedure. The first step is designed to provide an estimate of the GMO content of the lot based on a bulk sample. The second step is designed to provide an estimate of the associated sampling error, if required.

6.2.2 Step 1: Sampling of increments

Increments should be sampled following the technical principles given in ISO 6644:2002 as described in 6.3 and ISO 13690:1999 as described in 6.4.

The number of sampling points, where the increments for creating the bulk sample and the file increment samples are taken is defined according to the lot size. In case of lots from 50 to 500 tonnes, the size of the bulk sample should be 0.01% of the total lot size. In case of lots smaller than 50 tonnes, the size of the bulk sample should be 5kg. In case of lots larger than 500 tonnes, the size of the bulk sample should be 50 kg. (see Table 1).

At each sampling interval (systematic sampling) or sampling point (static sampling) an increment of 1kg should be collected and split into two portions of 0.5 kg: one to be used as an increment for the production of the bulk sample, the other to be stored as a file increment sample.

Note according to ISO 6644 the maximum bulk sample size is 100 kg and the increment size 1 kg, with a maximum lot size of 500 t. As a two step procedure is recommended to estimate the sampling error the increment of 1 kg is splitted in two leading to an increment size of 0,5 kg. The resulting bulk sample of 50 kg equals 0,01% of the maximum lot size as defined in ISO 6644. In order to maintain the sampling frequency at the same level, the bulk sample should be 0,01% independent of the lot size. For lots <50 t and lots >500 t the bulk sample size is fixed due to statistical and economical reasons, respectively.

Table 1 — Number of sampling points according to the lot size

Lot size in t	Size of the bulk sample in kg	Number of sampling points
≤ 50	5	10
50 to 500	0,01% of lot size	2 times bulk sample size in kg
≥ 500	50	100