codex alimentarius commission

FOOD AND AGRICULTURE ORGANIZATION OF THE UNITED NATIONS

WORLD HEALTH ORGANIZATION

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Agenda Item 3 **CX/MAS 02/3-Add.1**

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

Twenty-fourth Session
Budapest, Hungary, 18-22 November 2002

PROPOSED DRAFT GENERAL GUIDELINES ON SAMPLING

GOVERNMENT COMMENTS AT STEP 3

(Canada, Czech Republic, Hungary, South Africa, United States, IDF)

CANADA

Canada supports the Proposed Draft General Guidelines on Sampling and is of the opinion that the present document covers the essential elements and information needed to assist countries in their sampling programs. Therefore, Canada supports its advancement in the Codex step procedures.

CZECH REPUBLIC

General Comment

The Czech Republic recommend to complete guidelines with sampling plans for average content of characteristics in isolated lots, or if possible with sampling plans by variables. That type of sampling plans is most frequent and important in food inspection and control.

Individual comments

1. Page 2 - 3, Table of contents - correction/revision of the Contents is necessary, because of some errors (e.g. titles of some chapters in document text are missing in contents (e.g. 4.2 Single sampling plans...), some references to pages do not correspond to the respective text (e.g. Section One is not on page 5)

2. Some references to the chapters are not correct (only examples)

   (a) page 5, Table "For Chemical and Physical Characteristics" - references to chapter 2.4.3.4 are not correct (chapter does not exist)

   (b) pages 7-8, Table 1 - references to chapter 2.4.3.4 are not correct (chapter does not exist)

   (c) page 11, 2.2.1 General, 2nd para. - reference to Section 2.2.10 is probably not correct

   (d) page 15, 2.2.13 - reference to Section 2.2.11 is probably not correct

   (e) page 16, 2.2.15 - reference to Section 2.2.12 is probably not correct

   (f) page 34, 3.1, 2nd para. - reference to Section 2.2.10 for "switching rule" is probably not correct

3. pages 42, 46, 48, 53, 60, 63, Figures 5, 7, 8, 10, 11, 12, 13, 14, 15, 16 - some lines/ curves are not visible in printed document

HUNGARY

Remarks on the CX/MAS 02/3 „Proposed Draft General Guidelines on Sampling"

The proposed Draft is correct from statistical viewpoint. The remarks concern only for some practical application as well as for the form of tables and figures.

Page 2 and 20: The correct title of 2.5. is SINGLE SAMPLING PLANS.

Page 10: The enumeration of ANSI Standards is not necessary because such standards cannot be found on this list. The ISO/DIS Standards are not yet accepted.

Page 22: In the last box of the table the formula should be written:

$$\bar{x} < L + K\sigma \text{ , or } \quad \bar{x} > U - K\sigma$$

Page 25, similarly: The formula in the last box of the table should be written:

$$\bar{x} < L + Ks \text{ , or } \quad \bar{x} > U - Ks$$

Page 27 and 28: In our opinion, the comparison of the effectiveness of □ and *s*-method as well as the effectiveness of the attributive and variable plans is not recommendable. Namely, the application of □ and *s*-method as well as the use of attributive or variable plans should be determined by the nature of qualifying. The use of these sampling plans cannot be determined only from economical viewpoint.

Page 32: 2.5.3.1. This example for the application of the Standard (ISO 2859-0) is not recommendable even because of the too great sample size. Another example should rather be given.

Page 44 and 45: Table 11 should be written on the same page.

Page 45 and 46, similarly: Table 12 should be written on the same page.

SOUTH AFRICA

South Africa agrees with the general principles used in the guidelines and supports that the Codex product committees establish detailed and user-friendly sampling procedures for application at the implementation level.

However, we have noted that some references are used in the document that could not be found. They are:

Page 7, Table 1: Sections 2.4.2, and 2.4.3.4

Page 11: Sections 2.4.3.1, 2.4.3.2, 2.4.3.3, 2.4.3.4 and 2.4.3.5

Page 15: Section 4.1.1

Page 17: Section 2.4.3

Page 26: Sections 2.4.1.2.2 and 2.4.1.2.3

UNITED STATES

The proposed draft document appears to have addressed all of the previous concerns expressed by the Delegates and it has finally reached an acceptable level regarding its coverage of acceptance sampling topics. However, the document needs extensive work relative to proofreading, technical editing and style. Accompanying the following general comments is an annotated version of the document, which should be submitted as a conference room document for the drafting group's consideration.

General Comments

- There are a number of places (too numerous to mention) where references are made to clauses that are nonexistent or incorrect
- Numerous sentences and footnotes lack sufficient clarity and need rewording
- There are instances of misspelled words (e.g., StandardiZed, etc.)
- The document lacks consistency in naming or defining several sampling methods (e.g., s-method, method s, 's' method, etc.)
- There are several acronyms used to define acceptance number (e.g., c, Ac, etc)

- Definitions are needed to place practical meanings on several indefinite words (e.g., negligible, small, etc)
- Symbols used to define the sample mean are inconsistent (e.g., x, $\bar{x}$, etc)
- Tables and Figures need headings to introduce content
- Tables presenting sampling plans need to be restructured to make them appear less busy (e.g, in Table 9, et al, the instrance where the phrase "Probability to accept these lots" is used in four different columns could be replaced by a single line "Probability of Acceptance" extending across all four sampling plans.

INTERNATIONAL DAIRY FEDERATION (IDF)

SAMPLING PLANS FOR THE CONTROL OF THE COMPOSITION OF DAIRY PRODUCTS INSPECTION BY VARIABLES, KNOWN STANDARD DEVIATION

BACKGROUND INFORMATION

The IDF/ISO/AOAC Action Team on Statistics of analytical data had prepared the following document which is submitted as a contribution to discussion in the Codex Committee on Methods of Analysis and Sampling at the forthcoming meeting. The objective of this document is to provide some background information to familiarize the participants with statistical concepts, bearing in mind that the CCMMP will have to decide on the introduction of statistical sampling plans in the framework of control of dairy product composition. The members of CCMMP, who cannot be expected to be familiar with the statistical concepts, will have to be convinced that the use of such plans offers advantages compared with the status quo. It is to this group that the document is addressed.

Introduction

When controlling the composition of a dairy product, we are interested in the composition of a large quantity of product, e.g. a lot, or a consignment or even the total production of one week. A sample should therefore reflect the composition of the quantity submitted to control. The composition of dairy products normally shows a certain amount of variation. Statistical aspects are therefore to be taken into consideration, when drawing a sample.

In reality, sampling plans based on statistical considerations are normally not applied for official control. There is a simple reason : it is expected that sample sizes would be much higher than regarded as acceptable. Proposals to apply sampling plans must take this aspect into consideration.

In this paper an attempt is made to describe a procedure which should be regarded as acceptable and nevertheless, provides the necessary information.

Required information

Typical examples for the control of dairy product composition are the control of the fat, dry matter, protein or water content. Control results are in these and similar cases measurements results. The distribution of such results can very often be described by the arithmetic mean and standard deviation. Should it be possible to control arithmetic mean and standard deviation, adequate information on product composition would be available in all cases, where data distribution follows approximately a normal distribution.

Definition of limits

Assuming that information on arithmetic mean and standard deviation is available, how can compliance with a limit established by legislation be verified ? Before this question can be answered, a definition of limits is required.

Obviously, respect of the limit cannot mean that each part of the quantity to be controlled must comply, though limits are sometimes interpreted in this way (“100 % compliance” would be a requirement which cannot be controlled). It is therefore adequate to allow for a certain percentage of non-complying units. This concept is based on the (theoretical!) assumption that a very large sample (e.g. 1000 or 10.000 units) is taken from the quantity submitted to control. Under these circumstances the percentage of non-complying units would be nearly identical to the “true” percentage.

The limit could for example be set at 5 %, i.e. not more than 5 % of the units in the quantity to be controlled shall exceed the limit established by legislation (the legislator would have to decide whether 5 % or a higher or lower percentage is adequate). We can now define compliance with a limit.

An upper limit (U) is respected, if the following condition is fulfilled :
$u + 1.645\ \sigma_p \leq U$
u : true arithmetic mean
σ_p : true process standard deviation (a measure for the variation of product composition caused by a variation of processing conditions)
1.645 : a factor taken from statistical tables (on condition that the data distribution can be described as a normal distribution, we would expect that 5 % of the result exceed
$u + 1.645\ \sigma_{p)}$.

A lower limit (L) would be defined in an analogous way :
$u - 1.645\ \sigma_p \leq L$

It can be seen from the two equations that u must differ by at least $1.645\ \sigma_p$ from U or L; a lower difference means that more than 5 % of the units would give results which exceed the limit.

The approach described above enables us to define a limit precisely. However, how can we apply this concept in practice ? Unfortunately, it is not possible to determine the true arithmetic mean and the true process standard deviation. It will be demonstrated, that the concept developed here can form the basis of a very effective control procedure.

Elements of a control strategy

While it is not possible to determine the true arithmetic mean and the true process standard deviation of the parameter to be controlled, estimates can easily be obtained :

– $\bar{x}$ estimate for the arithmetic mean

– s_p estimate for the process standard deviation.

A random sample will provide this information. However, it has to be stressed that the determination of the process standard deviation requires sample sizes which would normally be regarded as unacceptably large. Consequently, in most cases the control of the process standard deviation will not be carried out by analyses in an official control laboratory. An appropriate procedure will be described in the next section.

Fortunately, only a limited amount of work is required to obtain an estimate for the arithmetic mean. In many cases it should be possible to analyse composite samples, thus reducing the amount of work considerably.

If for example a sample consisting of five sample units is taken and a composite sample can be made, just 1 analysis would be necessary for the determination of the arithmetic mean based on 5 sample units.

An example is used to describe the procedure for controlling the arithmetic mean and for data interpretation.

Assumptions : upper limit established by legislation : 16 %
Process standard deviation 0.15 %
Calculated upper limit for the arithmetic mean :
$16.00 - 1.645 \cdot 0.15 = 15.75$ %
When adjusting the process, the producer has to make sure that the arithmetic mean does not exceed 15.75 %.

Further assumption : 5 samples units are taken and analysed by the control authority.

Result of the analysis : $\bar{x}$ = 15.78 %

It would not be justified to conclude immediately that the upper limit for the arithmetic mean has been exceeded. As a consequence of process variation, the average composition of a sample will not be exactly the same as the average composition of the quantity to be controlled. The difference (sampling error) will decrease with the sample size.

The following formula allows to calculate the sampling error in cases where the process standard deviation is known :

95 % limits for the sample mean :

$u + 1.645\ \sigma/\sqrt{n}$ (upper limit)

$u - 1.645\ \sigma/\sqrt{n}$ (lower limit)

n : sample size

Assuming that the true arithmetic mean is exactly at the upper limit u = 15.75 %, we would obtain :
95% upper limit for a sample mean, sample size n = 5 :

$15.75 + 1.645 \cdot 0.15/\sqrt{5} = 15.75 + 0.11 = 15.84$ %

It can be seen that the control result obtained (15.78 %) is well below the upper confidence limit for the sample mean. Consequently, it cannot be concluded that the producer has adjusted the arithmetic mean at a too high level. In this case, there is no need to look at the lower confidence limit.
The above formula shows that the differences between u and the confidence limit become smaller, when the sample size is increased ($\sqrt{n}$ increases). The same control result based on a large sample size can thus lead to the conclusion that the upper limit for the arithmetic mean established by legislation has been exceeded.

It should be stressed that measurement error has to be considered as well, when evaluating control data. This aspect will be dealt with below.

USE OF AUTOCONTROL RESULTS

The sample sizes for obtaining reliable estimates for the process standard deviation are by far higher than normally acceptable for control authorities. It is therefore envisaged that the producer provides information on the process standard deviation based on his own control data (autocontrol results). On condition that the process standard deviation is stable over an extended period of time, a long-term process standard deviation (σ_p) can be calculated. σ_p is used by the control authority for the evaluation of the arithmetic mean determined in an official laboratory. By comparing the arithmetic mean reported by the producer with the arithmetic mean obtained by the control authority, information on the reliability if the reported arithmetic mean can be obtained.

An essential aspect is the control of the reliability of the reported process standard deviation. For the producer it may be tempting to report lower values for σ_p than found in reality.

There are several control options :

- comparison with the results obtained by other producers. Very low results are suspect and may require further investigations
- 2 samples are taken (instead of 1) and 2 composite samples are prepared. Larger differences than expected under the assumption that σ_p is correct, would indicate that the reported value may be too low (the suspicion may be confirmed by further analysis carried out in the future).
- A large number of sample units taken randomly is analysed in the presence of a control inspector in the dairy laboratory. A significantly higher result than expected would indicate that the reported value for σ_p is too low.

Consideration of analytical error, conclusion

When evaluating results, apart from σ_p the precision data of the method used by the producer and by the control authority are to be taken into consideration. Using all this information, it can be decided, whether

- The limit established by legislation has been significantly exceeded. In this case the conclusion would be drawn that the consignment controlled does not comply with the legal requirements
- The difference between the arithmetic mean reported by the producer and the arithmetic mean found by the control authority is significant. In this case the reliability of the results obtained by the producer may be questioned on condition that the official laboratory can prove by regular successful participation in proficiency tests that its results can be regarded as reliable.

(SUGGESTED) DRAFT STANDARD - MILK AND MILK PRODUCTS
APPLICATION OF SAMPLING PLANS FOR THE CONTROL OF THE COMPOSITION OF PRODUCTS IN THE PRESENCE OF MEASUREMENT ERROR
INSPECTION BY VARIABLES, KNOWN PROCESS STANDARD DEVIATION

Background information

The IDF/ISO/AOAC Action Team on Statistics of analytical data has prepared the following document which is submitted as a contribution to the Codex Committee on Sampling and Analysis at the forthcoming meeting. The suggested draft standard shows how the relative complex approach discussed in CCMAS can result in a fairly simple approach. It has, in fact, been successfully applied in practice.

We draw your attention to two aspects:

- The amount of work can be reduced considerably by analysing composite samples (this is possible in many cases). A requirement to analyse sample units individually in order to determine the sample standard deviation results in a considerable amount of work, because large samples would be needed This is the main reason why an approach based on known standard deviations has been proposed.
- Measurement uncertainty is taken into consideration, when applying the proposed plan.

Introduction

Inspection by variables is a method which consists in measuring a quantitative characteristic for each item of sample taken from a population. The acceptability of the population is established statistically by evaluating the difference between the limit for a compositional characteristic established by legislation/ a contract and the arithmetic mean of the control result obtained with a random sample.

Scope
This International Standard describes the application of sampling plans for the inspection by variables of milk and milk products. It is intended for use by contract partners (buyers) or control authorities under the following conditions:
a) The quantitative characteristic to be controlled is approximately normally distributed and processing conditions are stable (under statistical control)

b) The process standard deviation, describing the variation of the quantitative characteristic as a consequency of variations of the processing conditions, is known

c) Upper or lower specification limits are prescribed by legislation or contracts.

d) Repeatability and reproducibility standard deviation of the analytical method used for compliance testing are known.

This International Standard does not apply in the case of examination of microbiological defects.

References
See IDF Standard 136A : 1992

Definitions
See IDF 136A : 1992

Compliance with an upper specification limit U or a lower specification limit

A consignment complies with an upper specification limit U, if the following condition is fulfilled :

$$\mu \leq U - z\sigma_p \qquad (1)$$

A consignment complies with a lower specification limit L, if the following condition is fulfilled :

$$\mu \geq L + z\sigma_p \qquad (2)$$

Where
μ is the true arithmetic mean of the characteristic to be controlled
z is the z-value corresponding to a given probability (to be taken from statistical tables; see below)

σ_p is the long-term process standard deviation of the characteristic to be controlled.

The information on σ_p is provided by the producer
σ_p does not include the repeatability standard deviation σ_r. It is obtained by the equation :

$$\sigma_p = \sqrt{\sigma^2 - \sigma_r^2}$$

σ : process standard deviation including the repeatability standard deviation σ_r
σ is used by the producer to establish compliance with the requirements. However, he reports σ_p to the control authority.

In cases where the measurement variance is [≤ 15 %] of the total variance, measurement variance has not to be taken into consideration when determining σ_p.

In cases where the process variance is [≤ 15 %] of the total variance, there is no need to report σ_p. In this latter case σ_p in equations (1) and (2) is replaced by σ_r.

Fulfillment of the compliance conditions specified above means that the probability to obtain a result not exceeding the specification limit is at least x %. It is up to the competent authority to specify compliance conditions characterized by different probabilities (e.g. 90 %, 95 % or 99 %, corresponding to z = 1.282, z = 1.645 and z = 2.326, respectively) in legislation. In contracts the probability agreed upon by both parties should be specified.

Determination of the long-term process standard deviation σ_p
The producer takes an adequate number of sample units from each lot/per production day and determines σ_p. A total number of at least [150] results obtained with at least [10] different lots/on at least [10] different production days forms the basis for the determination. On condition that the variation between lots/production days is acceptable, the weighted mean of the variances is determined. The square root of the weighted mean is the provisional long-term process standard deviation, used to start the control procedure.
The validity of the reported σ_p value is regularly checked by the producer and adjustments are made, if need be.

Compliance control
The competent authority takes a random sample of n sample units (n to be determined by legislation or to be agreed upon by the contracting parties). Where possible a composite sample/composite samples can be prepared and analyzed. The consignment shall be considered to comply with the requirements, if the arithmetic mean of the results does not exceed μ. Compliance with the requirements is tested in cases, where $\bar{x}$ exceeds μ, as follows :

$\bar{x} \leq \mu_u + 1.645\ \sigma_{\bar{x}}$ (upper limits)

$\bar{x} \geq \mu_L - 1.645\ \sigma_{\bar{x}}$ (lower limits)

μ_u, μ_L : upper (lower) limit for the arithmetic mean, determined using equation (1) and (2), respectively

$$\sigma_{\bar{x}} = \sqrt{\frac{\sigma_p^2}{n} + \sigma_L^2 + \frac{\sigma_r^2}{n_1}}$$

n_1 = number of composite samples
σ_L : between-laboratory standard deviation

$$\sigma_L = \sqrt{\sigma_R^2 - \sigma_r^2}$$

σ_R : reproducibility standard deviation
σ_r : repeatability standard deviation

Control of the reported process standard deviation σ_p
There are several options:

a) comparison with σ_p reported by other producers :

If the reported σ_p-value is low compared to values reported by several other producers for the same product, an investigation should be made. The same conclusion should be drawn, if there is evidence that the information provided by a producer is unreliable (example : reported $\bar{x}$-values not compatible with $\bar{x}$-values found by the official control laboratory).

b) 2 composite samples are taken and analyzed :

Instead of 1 composite sample, 2 composite samples from a consignment are analyzed and the results compared. The 2 results $\bar{x}_1$ and $\bar{x}_2$ are evaluated as follows:

$$z = \frac{\bar{x}_1 - \bar{x}_2}{\sigma_d}$$

$$\sigma_d = \sqrt{2\sigma_r^2 + 2\frac{\sigma_p^2}{n}}$$

Disputes can be avoided by analyzing each composite sample in duplicate (triplicate) thus demonstrating that the laboratory worked in compliance with the repeatability criteria. In these cases σ_r^2 has to be divided by 2 and 3, respectively.

z-values exceeding 1.96 indicate that the reported σ_p-value is too low.

c) Analysis of a large sample in the dairy control laboratory under supervision

A large sample taken randomly from the production line or store is analyzed in the presence of a control inspector. A sample standard deviation significantly exceeding σ_p (significance tested using the Chi^2-test) indicates that the reported value for σ_p is too low.

Abuse of tolerances

When the arithmetic mean of the control results exceeds the upper limit for μ, tolerances are applied for the final evaluation of results (see section 6). There is a risk that these tolerances are abused by the producer. Results obtained in two consecutive controls exceeding the upper limit for μ, but regarded as complying, when the tolerance is applied, should lead to the following actions:

The arithmetic mean of the control results obtained by the producer is compared with the arithmetic mean obtained by the control authority. The following test is applied :

$$z = \frac{\bar{x}_1 - \bar{x}_2}{\sigma_d}$$

$\bar{x}_1$: arithmetic mean of the results obtained by the producer

$\bar{x}_2$: arithmetic mean of the results obtained by the control authority

$$\sigma_d = \sqrt{\sigma_{L1}^2 + \frac{\sigma_{r1}^2}{n_1'} + \frac{\sigma_p^2}{n_1} + \sigma_{L2}^2 + \frac{\sigma_{r2}^2}{n_2^1} + \frac{\sigma_p^2}{n_2}}$$

Methods applied in the producer laboratory :

σ_{L1} : between-laboratory standard deviation

$\sigma r1$: repeatability standard deviation

n_1' : number of composite samples analysed

n_1^1 : sample size
Method applied by the control authority :
σ_{L2} : between-laboratory standard deviation
σ_{r1} : repeatability standard deviation
n_2^1 : number of composite samples analysed
n_2 : sample size

If z exceeds 1.96, there is a method bias. Adjustments/corrections are necessary in the dairy control laboratory. This conclusion can only be drawn, if the laboratory of the control authority has regularly and successfully participated in inter-laboratory comparisons (proficiency tests).
Where no significant difference is found, further actions depend on the result obtained with the next consignment.

Results not exceeding μ will lead to normal control actions in the future, while results in the "grey area" have the consequence that a tightened control is immediately carried out. When the next consignment from the suspect producer arrives, the sample size and the number of composite samples are increased.

Should the producer pass this test, the procedure is continued until results are in the normal range.