codex alimentarius commission

FOOD AND AGRICULTURE ORGANIZATION OF THE UNITED NATIONS

WORLD HEALTH ORGANIZATION

JOINT OFFICE: Viale delle Terme di Caracalla 00153 ROME Tel: 39 06 57051 www.codexalimentarius.net Email: codex@fao.org Facsimile: 39 06 5705 4593

Agenda Item 3b) **CX/MAS 07/28/4**

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING
Twenty-eighth Session
Budapest, Hungary, 5 – 9 March 2007

DRAFT GUIDELINES FOR SETTLING DISPUTES OVER ANALYTICAL (TEST) RESULTS

GOVERNMENT COMMENTS AT STEP 6

ARGENTINA (English version)

Argentina appreciates the possibility of commenting again on this document. Argentina considers that the scope of the document should be restricted exclusively to disputes on analytical results in the area of chemistry or physics / chemistry, as it considers that microbiological analysis should be specifically excluded from the scope of this guide. The opinion of Argentina is based on the following concepts:

- When samples are re-analysed (be it test sample or re-sampling of the lot) to confirm the initial result of a food sample in which a pathogen is detected, the result obtained with the second analysis often differs from the original one.
- This may happen even when the size of the sample analysed is increased in the second sampling.
- If laboratory and sampling errors are certainly possible, the possibility that the distribution of the microorganism should be heterogeneous[1] cannot be discarded (random sampling cannot detect pathogenic microorganisms when the distribution is heterogeneous), or that the prevalence[2] of contamination should be low or that the pathogen should have lost its viability between the first and the second analysis.
- Experience with two class sampling plans by attributes for pathogens shows without doubts that heterogeneous distribution or low prevalence of these microorganisms can justify discrepancies between initial and subsequent results, for confirmation.

Reference: ICMSF: Microorganisms in Food 7 (microbiological analysis in food safety management).

ARGENTINA (version en español)

Argentina agradece la posibilidad de realizar nuevamente comentarios al presente documento Argentina considera que el alcance de este documento debería restringirse exclusivamente a las disputas sobre resultados analíticos en el ámbito de la Química o Fisicoquímica, ya que considera que los análisis microbiológicos deben ser puntualmente excluidos del alcance de esta guía. La opinión de Argentina se sustenta en los siguientes conceptos:

- Cuando se reanalizan muestras (ya sean contramuestras o remuestreo del lote) para confirmar el resultado inicial de una muestra de alimento en que se detectó un patógeno, es frecuente que de este segundo análisis no se vuelva a obtener el resultado original.
- Esto suele suceder aún cuando en el segundo muestreo se aumente el volumen de la muestra analizada.

[1] Heterogeneous distribution: contamination occurs in a determined period of time during production. This type of contamination occurs due to contaminated equipment, to the working environment, to the operators or maintenance personal, among other examples

[2] Prevalence: frequency with which multiple simples of a specified product give a positive result in an analysis

- Si bien son factibles los errores de laboratorio y de muestreo, no se puede descartar que la distribución del microorganismo en el alimento sea heterogénea[1] (un muestreo aleatorio, cuando la distribución es heterogénea, no puede detectar al microorganismo contaminante), que la prevalencia[2] de la contaminación sea baja o que el patógeno haya perdido viabilidad entre el primero y segundo análisis.
- La experiencia con planes de muestreo de atributos de dos clases para agentes patógenos demuestra sin lugar a dudas que las distribuciones heterogéneas o la baja prevalencia de estos microrganismos pueden justificar las discrepancias entre los resultados iniciales y los siguientes, de confirmación.

Referencia: ICMSF: Microorganismos de los Alimentos 7 (Análisis Microbiológico en la Gestión de la Seguridad Alimentaria).

AUSTRALIA

SECTION 3. PROCEDURE

Australia believes that the flow chart supplied in Section 3. Procedure is of little use in its current format, but that a properly constructed flow chart would be most a useful inclusion in this paper. Consequently, Australia has provided an alternative flow chart (see attachment in ANNEX) for consideration.

SECTION 3.3 – STEP 3: NEW ANALYSES ARE CARRIED OUT

Australia believes that the text relating to the two scenarios listed needs to be further clarified. For example, Point 1 currently does not include all possibilities outlined in Point 3 of the 'Step 3: New Analyses are Carried Out', prerequisites regarding analyses in one laboratory i.e. the analyses could also have been performed in a third laboratory chosen on the basis of consensus or selected by the competent authority of the importing country. Given that an agreement must have already been reached as a prerequisite to this option if the shared sample is only to be analysed in one laboratory i.e. by either one of the two laboratories in the presence of the other; or a third laboratory selected by consensus of the exporting and importing countries; or by a laboratory selected by the competent authority of the importing country, Australia suggests that the text be revised to simply state "***analyses are performed in one laboratory and the new results are used to assess conformity***."

In Point 2 it is not clear if the statement "If the new results are in agreement" relates to the new results being in agreement with each other or the original result of the shared sample. There are a number of possibilities here e.g. if Laboratory A's reserve sample is analysed by both laboratories and:

- the new results from both labs are in agreement with each other and the original result - dispute is over, Laboratory A's results used to assess conformity, or;
- the new results from both labs are in agreement with each other but different to original result of Laboratory A - a decision needs to be made to discard original result and assess conformity on the basis of the new results, or;
- the new results from both labs are in agreement with each other, different to original result of Laboratory A but agree with original result of Laboratory B - dispute over and Laboratory B's results used to assess conformity, or;
- Laboratory A's new result agrees with its original result but not with the result of Lab B -check for bias (i.e. go to Option 3A), or;
- Laboratory B's new result agrees with Laboratory A's original result but not with Laboratory A's new result - indicates problem with Laboratory A's new result, assess conformance on Laboratory A's original result.

While a number of these scenarios are less likely, they are possible. Consequently, Australia suggests the text of Point 2 be changed to "***the two laboratories analyse the samples separately: if an agreement can be***

[1] Distribución heterogénea: la contaminación sólo se produce en un determinado segmento de tiempo de la producción. Este tipo de contaminación se produce por un equipo contaminado, por el ambiente de trabajo, por los operadores o personal de mantenimiento, entre otros ejemplos.
[2] Prevalencia: frecuencia con que muestras múltiples de un producto determinado dan resultado positivo en un análisis.

reached on the basis of the new results, the dispute is settled. If no agreement is reached, resolution of the dispute may be sought by proceeding to step 4".

BRAZIL

Page 3 – Item 3.1 – 3º paragraph

Brazil proposes to change the text as follows:

From:

In other cases, the ANNEX suggests a simple procedure, based on the Horwitz's model, to implement this criterion and resolve the dispute. When available or recognised, other models than Horwitz's could be used.

To:

In other cases, the ANNEX suggests a simple procedure, based on the Horwitz's model, to implement this criterion and resolve the dispute. When available or recognised, other models than Horwitz's such as Thompson's model could be used.

Justification: Ratifying the quotation of Thompson model that appears in the figure 2, in the table 1 of page 8 and in the references.

Page 6

$$T = \frac{Y1 + Y2}{2}$$

$Y1$, $Y2$ and T must be dimensionless, for example, for 1 ppm (mg/kg) $T = 10^{-6}$

Justification: an example, to be clear.

$$\Delta \max\ (\%) \leq \frac{0.0566 \times T^{0.8495}}{T} \times 100$$

Justification: coherence with the described in the heading of table 1.

Page 8

Table 1.

There is a little error of figure in the last expression for s_R. The correct would be:
$s_R = 0.22 \times T$

CUBA (version in English)

1) It is proposed to limit the applicability of the document only to physical, chemical and physic/chemical tests, excluding microbiological tests, which are not ruled by the same principles regarding the requisites for sampling as the above tests. The propagation of microorganisms follows very specific distribution patterns, closely related with storage and preservation of the sample. Normally in microbiological tests it is not easy to obtain identical replicas, or within acceptable limits, of the original test. This may be due to factors which are outside the sampling procedure itself, the distribution of microorganisms does not have to be necessarily homogeneous in all units from the same food lot or may vary according to its state of development or inhibition throughout the period of time considered

2) The document is acceptable without other amendments and it is considered very useful in the development of trade relations.

CUBA (version en español)

1. Se propone limitar la aplicabilidad del documento sólo para los ensayos físicos, químicos y físico-químicos, excluyendo los ensayos de tipo microbiológicos, los cuales no se rigen por los mismos principios en cuanto a los requisitos para la toma de las muestras que el resto de los ensayos antes

mencionados. La propagación de los microorganismos ocurre según distribuciones muy específicas, estrechamente relacionadas con las condiciones de almacenamiento y conservación de la muestra. En los ensayos microbiológicos normalmente no es fácil obtener réplicas idénticas, o dentro de los límites aceptables, de un resultado de ensayo original. Esto puede deberse a cuestiones ajenas al procedimiento de muestreo propiamente, la distribución de los microorganismos no tiene por qué ser necesariamente homogénea en todas las unidades procedentes del mismo lote del alimento o pueden variar su estado de desarrollo o de inhibición con el transcurso del tiempo.

2. Se acepta el documento propuesto sin otras modificaciones y se considera de gran utilidad en el desarrollo de las relaciones comerciales.

IRAN

1- SCOPE

Iran proposes that the first sentence from the third paragraph of the scope is read: "these guidelines do not adress questions of sampling".

2- PREREQUISITES

Compliance with "quality assurance provision", stated in the first paragraph of the prerequisites has not been defined, therefore may cause fuerther disputes by itself. Iran proposes that this compliance should be referred to an internationally agreed or well defined quality assurance guidelines to prevent different interpretations.

Iran further suggests that the first paragraph read:

"laboratories comply with quality assurance provision **and/or** with the codex Guidelines for the Assessment of the Competence of Testing Laboratories Involved in the Import and the Export of Food"

3- Footnote 5 on page 4
Iran proposes that ambiguous terms such as "several samples" and the "appropriate number" should be well defined.

MALAYSIA

1. SCOPE

Malaysia proposed to include the phrase *"**underlying these guidelines***" after the word assumption and omit the words *"**that when**"*. Malaysia also would like to change the position of the word *"**made**"*, which occurred after the word ***"results"***, to between the word ***"assessment"*** and the word ***"based"***.

This paragraph is to read:

> *"The basic assumption **underlying these guidelines** is the assessment **made** based on test results in the importing country disagrees with the assessment made by the exporting country."*

3.1. - STEP 1: THE ANALYTICAL RESULTS ARE COMPARED USING THE REPRODUCIBILITY LIMIT

Para. 3 of the ANNEX:

Malaysia proposed to include the phrase "***or if there are two published reproducibilities from two different methods used by the two laboratories***" after the word reproducibility.

The sentence should read as:

*"If there is no published reproducibility **or if there are two published reproducibilities from two different methods used by the two laboratories**, it is possible to use the model of Horwitz to calculate the limit of reproducibility as:"*

3.2. - STEP 2: THE RESULTS AND PROCEDURES OF THE LABORATORY OF THE EXPORTING COUNTRY AND ITS COUNTERPART IN THE IMPORTING COUNTRY ARE COMPARED

1) Malaysia proposed to rearrange the sentence "***validation status of the methods of analysis used (including method specific sampling and preparation procedures),"*** in the first bullet.

The sentence in the first bullet should read:

"validation status of the methods of analysis used (including specific sampling ***method*** *and* ***preparative*** *procedures),"*

2) Malaysia noticed a typographical error in the sentence of :

a) the fifth bullet. The fullstop mark at the end of the sentence *"performance in relevant proficiency testing or collaborative studies****.****"* should be changed to a *"comma"* and the word *"****and****"* is added at the end of the sentence.

The sentence should read:

*"performance in the relevant proficiency testing or collaborative studies****, and****"*

b) the last bullet. The word "**and**" at the end of the sentence *"official accreditation status of the laboratories and"* should be deleted and a "*fullstop*" should be placed at the end of that sentence.

As such, the sentences will read as follows:

*"official accreditation status of the laboratories****."***

3.3. - STEP 3: NEW ANALYSES ARE CARRIED OUT

Prerequisites

Malaysia felt that the sentence of the third bullet is rather ambiguous and would like to seek clarification on:

(a) the priority of the three options given. Should the options be considered according to the sequence they were given or the degree of importance?

(b) the decision on the choice of options between the first option and the second option given. Is it a choice which will be decided by consensus or by the importing country? This is because for the third option it was clearly stated that a third laboratory may be selected by consensus, or, failing that, by the competent authority of the importing country.

Available approaches

C. – ANALYSES OF RESERVE SAMPLES

Subpara (2):

The word *"****out****"* should be added after the word *"carry"* and the *"****:****"* after the word *"separately"* should be changed to a *"****.****"*.

The sentence should read:

"(2) the two laboratories carry ***out*** *analyses separately****.*** *If the new results are in agreement, the dispute is settled. If no agreement is reached, resolution of the dispute may be sought by proceeding to step 4."*

Malaysia would like to seek clarification on why the options listed under C. – ANALYSES OF RESERVE SAMPLES (where 2 options were stated) do not tally with the options listed under Prerequisites no. 3 (where 3 options were stated).

ANNEX

Table 1. Published recognized models

The information in the second column "Range (dimensionless)" and the third column "Equation of s_R" of the table (shown below) is quite ambiguous.

Name	Range (dimensionless)	Equation of s_R
Horwitz [1]	**10^{-1} to 1.2 10^{-7}**	$s_R = 0.02 \times T^{0.8495}$
Thompson [2]	**> 1.38 10^{-1}**	$s_R = 0.01 \times T^{0.5}$
	1.38 10^{-1} to 1.2.10^{-7}	$s_R = 0.02 \times T^{0.8495}$
	< 1.2.10^{-7}	**$s_R = 0.02 \; T$**

The information in the second and third columns of the table should be rewritten as follows:

Name	Range (dimensionless)	Equation of s_R
Horwitz [1]	10^{-1} to 1.2 **x** 10^{-7}	$s_R = 0.02 \times T^{0.8495}$
Thompson [2]	> 1.38 **x** 10^{-1}	$s_R = 0.01 \times T^{0.5}$
	1.38 **x** 10^{-1} to 1.2 **x** 10^{-7}	$s_R = 0.02 \times T^{0.8495}$
	< 1.2 **x** 10^{-7}	$s_R = 0.02$ **x** T

NEW ZEALAND

GENERAL COMMENTS

New Zealand believes that this document will provide valuable guidance to governments. We recommend however some changes as outlined below.

1. SCOPE

We recommend that footnote 3 should include further items that are outside the scope of the guidelines, so that the scope is as clear as possible. The footnote would then read:

> Possible reasons for disagreement may include one or several causes such as: the existence, appropriateness and statistical validity of the sampling plan used to assess the product; the allowances made for normal measurement error and within-lot product variation; differences in physical sampling procedures; differences in composition of the samples tested due to product inhomogeneity or changes occurring during storage and/or transport of the product; differences in the methods of analysis or the laboratory performance; differences in the specification or results; differences in the expression of results (corrected for recovery, etc).

2. PREREQUISITES

Second bullet

(a) We recommend a change to allow for more than one sample, as agreed at the last meeting.
(b) There are marked advantages of having the two duplicates of the contentious sample available for dispute resolution. New Zealand therefore recommends that three-way splitting of the sample or samples used in the test should be strongly encouraged by the guidelines, and should become part of the normal machinery of compliance testing.

With these changes the second bullet will read:

- at least one representative analytical laboratory sample from the same food consignment has been taken in accordance with established sampling plans and/or good sampling practices, where applicable; the laboratory sample or samples have been split for the purposes of analysis and for confirmatory analysis (reserve sample); the reserve samples should be kept in a satisfactory condition for the appropriate length of time. A three way split is strongly recommended where it is possible.

A new third bullet

New Zealand proposes that a new third bullet point should be included to clarify the scope of disputes that may be resolved by the Guidelines, as follows:

- The parties to the dispute either
  a) agree that the only question at issue is the validity of the analytical test results, or
  b) recognize that when the disagreement over analytical test results has been resolved by this procedure, other questions may remain to be decided.

3.1. STEP 1: THE ANALYTICAL RESULTS ARE COMPARED USING THE REPRODUCIBILITY LIMIT

(a) We recommend that this section should include a paragraph to explain that results are compared on the basis of analysis of duplicate samples.

(b) New Zealand strongly objects to the recommendation that the contentious result is averaged with the exporting laboratory's result. This is recommended in the procedure even when there is no reason to suppose that there is anything wrong with the exporting laboratory's result, and the two sets of results may be close enough to be consistent, allowing for normal measurement error. This is very much to the benefit of the exporter, and will significantly reduce the probability of non-compliant product being failed, particularly when the exporter's result is not based on a duplicate of the contentious sample. It also goes against standard practice, which is not to overturn a result unless there is evidence to do so.

(c) The procedure should include guidance for the situation where the laboratories have used different methods of analysis.

(d) The procedure should also include guidance for the situation where the importing country tests more than one sample and uses the mean to determine compliance.

To take account of these points, we suggest that 3.1 (Step 1) should be worded as follows:

> The importing country will supply to the exporting country one of the reserve sample(s) for analysis by a suitably qualified laboratory of its choice.
>
> When the difference between the two test results is within the reproducibility limit as defined below, the original assessment of the lot by the importing country shall stand.
>
> When both laboratories have used the same method of analysis and published reproducibility limits exist for the method, these limits should be used.
>
> When the laboratories have used different methods, but published reproducibility limits exist for both methods, the root-mean-square average of the two reproducibility limits should be used. However, if the compliance test used specified a particular method, with published reproducibility limits, and the laboratory in the importing country used a different method and this did not of itself invalidate the test, then the reproducibility of the specified method should replace that of the method actually used in the test.[1]

In other cases, the ANNEX suggests a simple procedure, based on the Horwitz model, to implement this criterion and resolve the dispute. When available or recognised, other models than Horwitz's could be used.

In cases where the test used by the importing country involved more than one sample, and the mean analyte level is the basis for assessment of the lot, the difference between the mean analyte levels obtained by each of the two laboratories should be compared using the reproducibility of a mean of several samples, as given in the Annex.

If results are outside the reproducibility limit, the attempt to resolve the dispute should proceed to step 2.

In case these models cannot be applied, the attempt to resolve the dispute should proceed directly to step 2.

(Footnote) [1] This envisages the possibility that under some conditions a laboratory may be permitted to substitute an alternative method of analysis for the one specified in the compliance test, In a dispute in which this has occurred, the exporting country should not be disadvantaged by the use of a test of reduced precision; equally the importing country should not be disadvantaged by use of a method of better precision.

3.2. STEP 2: THE RESULTS AND PROCEDURES OF THE LABORATORY OF THE EXPORTING COUNTRY AND ITS COUNTERPART IN THE IMPORTING COUNTRY ARE COMPARED

If samples have been split three ways, as recommended above, and duplicates remain for analysis, then New Zealand recommends a new step 3 that could be used in place of step 2. An introductory sentence would be needed to introduce this possibility as follows:

If reserve samples remain after Step 1 has been carried out, the two competent authorities involved may by agreement omit this step and proceed directly to Step 3. Otherwise, in accordance with relevant Codex Guidelines …

NEW STEP 3

Rather than the protracted and possibly inconclusive procedures suggested at steps 2 and 4 to determine which laboratory seems most likely to be closest to the true value, the parties to the disputes should at least have the option, if the dispute is not resolved at step 1 (that is if the difference is beyond the reproducibility limit), of having the matter decided by analysis of a third sample (part of an original three-way split of the sample on which the finding of non-conformity was based), preferably at a different laboratory and preferably with a sample of reference material analysed in parallel to enable a correction for laboratory bias on this third analysis to be made. Although this may seem rather arbitrary compared to the careful deliberations suggested in the revised guidelines, it has the merit of concentrating directly on the real question at issue – the true analyte concentration in the contentious sample.

We therefore propose the following wording for a new step 3 to be inserted following step 2:

3.3 – Step 3: The dispute is settled by analysis of the remaining reserve sample(s)

Where a second reserve sample is available, this should be analysed by a suitably qualified laboratory agreed on by the two countries, and a final assessment of conformity based on the result. The original result and the results from the first reserve tested under Step 1 should be discarded. If possible this laboratory should be different from the two laboratories whose results were compared in step 1. It is highly desirable that a Certificated Reference Material be analysed in conjunction with this under identical conditions if possible, and a correction for bias made to the results, according to the formula $y_C = y - (y_{ref} - x_{ref})$ where

y_C is the corrected result to be used in assessing compliance

y is the laboratory result on the third duplicate

y_{ref} is the laboratory result on the certified reference material

x_{ref} is the certified analyte concentration for the certified reference material.

If a second reserve sample is not available resolution of the dispute may be sought using Step 4. Even though the dispute may have been settled by use of Steps 1 to 3, investigations along the lines

suggested in Step 4 may still be of value, particularly if in the past disputes between the two countries going beyond Step 1 have occurred frequently.

ANNEX

(a) If the recommended changes to Step 1 are accepted, the paragraph "Measurement Uncertainty of the Mean" could be deleted.
(b) If the suggested change to allow for comparison of sample means between the two laboratories to allow for the compliance test being based on the mean of several samples is adopted, a formula for calculating reproducibility is needed, as follows:

Reproducibility limit for the mean of several samples:
This is given by

$$2.83 \times \sqrt{s_R^2 - s_r^2 + (1/n)s_r^2}$$

where

n is the number of samples over which the mean is taken
s_R is the reproducibility standard deviation
s_r is the repeatability standard deviation.

If there is no published repeatability for the method, or if the Horwitz model is used, s_r could be taken as approximatel

NORWAY

Norway appreciates the work done in the CL 2006/47-MAS. This document will be an important tool whenever there is a need to settle a dispute over analytical test results in worldwide trade.

However, we have a few suggestions of amendments we would like to provide for consideration.

1. Scope, Second paragraph. The sentence should be changed to:

The basic ~~assumption~~ ***application for using this guideline*** is ~~that~~ when the assessment based on test results made in the importing country ~~disagree~~s ***does not conform*** with the assessment made by the exporting country

2. Prerequisites
The first sentence should be changed to:
The ***specific*** procedure described in these Guidelines may ~~only~~ be used when

2. Prerequisites , first bullet, in the first sentence.
Insert "criteria specified in".

- laboratories comply with quality assurance provisions and with ***criteria specified in*** the Codex *Guidelines for…….*

2. Prerequisites
Insert a new paragraph at the end of section 2:
However, the list of information to be shared between competent authorities of the importing and exporting country as specified in article 3.2 should, when relevant, be applied in disputes on laboratory results between competent authorities of the importing and exporting country. Furthermore, the procedure described in article 3.4 analysing new samples from the consignment is also a relevant tool for solving various kinds of disputes.

Justification for inserting the new paragraph

Article 17 of "Guidelines for the Exchange of Information between Countries on Rejection of Imported Food – CAC/GL 25-1977" provides some guidance on exchange of information when disputes over analytical results occur. However, it is Norway's experience that importing and exporting countries often are reluctant to provide relevant information to each other on the analytical methods employed and the results obtained. Further guidance is therefore needed.

Chapter 3.2 of the draft "Guidelines for settling disputes over Analytical (Test) Results" provides a detailed list of such information to be exchanged if requested. However, the present text of chapter 2 restricts when this information can be requested: The laboratories have to comply with quality assurance provisions and with the Codex Guidelines for the Assessment of the Competence of Testing Laboratories involved in the Import and the Export of Food (CAC-GL 27).

Chapter 3.2 is valid outside the limited context of the document, and should therefore be made more universal. In the same way the method provided in article 3.4, namely by analysing new samples from the consignment, is a method that may also be applied more generally to resolve disputes.

The proposed new paragraph ensures that these parts of the guideline can be applied more generally in accordance with the texts of CCFICS.

Chapter 3, Flow Chart.
The flow chart provided by Australia has been brought to our attention. We think it is more instructive than the flow chart that's presented in the CL 2006/47. Norway therefore suggests the Australian flow chart to be included in the guideline instead of the one in the present draft.

Chapter 3.1, third paragraph: insert "possibly" (based on the Horwitz's model, to implement this criterion and ***possibly*** resolve the dispute~~)~~

Chapter 3.2, first bullet - Insert the words in bold cursive:

- ***description, reference to publication and*** validation status of the methods of analysis used (including method specific sampling and preparation procedures),

Chapter 3.2, last bullet. Delete the last world

- official accreditation status of the laboratories ~~and~~

Chapter 3.2, at the end: Add a new last paragraph:
If no reserve sample is available, proceed to step 3- A and/or to step 4

Chapter 3.4 add a new sentence at the end:
Agreement can be made on performing the sampling and analyses in the presence of both parties involved.

Comments of Australia
ANNEX

DISPUTE RESOLUTION