codex alimentarius commission

FOOD AND AGRICULTURE ORGANIZATION OF THE UNITED NATIONS

WORLD HEALTH ORGANIZATION

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Agenda Item 3b) **CX/MAS 08/29/4-Add.1**

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING
Twenty-ninth Session
Budapest, Hungary, 10 – 14 March 2008

CRITERIA FOR EVALUATING ACCEPTABLE METHODS OF ANALYSIS DRAFT GUIDELINES FOR SETTLING DISPUTES OVER ANALYTICAL (TEST) RESULTS

GOVERNMENT COMMENTS AT STEP 6

BRAZIL

Revised version of the Flowchart presented in CX/MAS 08/29/4. See Annex 3.

HUNGARY

Revised version of the Draft Guidelines

The Government comments at Step 6 made by Argentina, Australia, Brazil, European Community and New Zealand were considered and used for the revision of the Draft Guidelines for Settling Disputes over Analytical (Test) Results. As Flowchart (see Annex 1) the former Australian proposal was included. In addition to the proposed corrections the Draft Guidelines were extended with 2 additional versions proposed by the European Community (a part of the Room Document CRD 19) and by New Zealand which should be discussed in the Session.

DRAFT GUIDELINES FOR SETTLING DISPUTES OVER ANALYTICAL (TEST) RESULTS (At Step 6 of the Procedure)

1. SCOPE:

These guidelines provide guidance to governments on the procedures to resolve disputes which arise between food control authorities about the status of a food consignment[1], when the test results by the laboratory[2] in the importing country disagree with test results by the laboratory in the exporting country over the same lot[3].

The basic assumption is that when the assessment based on test results made in the importing country disagrees with the assessment made by the exporting country.

These guidelines only address disputes related to methods of analysis or laboratory performance and do not address questions of sampling. It is recognised that disputes may arise from other cause(s), which should also be investigated[4].

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[1] Status of the food consignment depends on the "interpretation" of the test result(s), in the light of measurement uncertainty, sampling error and the closeness of those test results to the limit. It could still be that the results do not differ by an amount which is significant, but nevertheless one result indicates conformity, but the other result does not.

[2] For the purpose of these guidelines, the word "laboratory" applies to both official and officially recognised laboratories. An official laboratory would be a laboratory administered by a government agency having jurisdiction empowered to perform a regulatory or enforcement function or both. An officially recognised laboratory would be a laboratory that has been formally approved or recognised by a government agency having jurisdiction.

[3] As defined in the General Guidelines for Sampling (CAC/GL 54 -2004)

[4] 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

These guidelines do not cover microbiological test results[5].

2. PREREQUISITES:

The procedure described in these Guidelines may only be used when:

- laboratories 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)*; and the laboratories have been designated by their respective Competent Authorities in both the importing and exporting countries;
- at least, one representative analytical laboratory sample from the same food lot has been taken by each Competent Authority 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 sample has been kept in a satisfactory condition for the appropriate length of time.

3. PROCEDURE: FLOWCHART (see Annex 1)

The settlement of the dispute without new analysis or sampling operations should be the preferred option as far as possible.

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

When the difference between the test results are within the existing reproducibility limit, the mean value of the test results of the 2 laboratories should be used to assess conformity, taking into account measurement uncertainty of the mean (see ANNEX 2 for definition).

When both laboratories have used the same method of analysis and published reproducibility limits exist for the method, these limits should be used.

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.

If results are outside the reproducibility limit, or these models cannot be applied, the attempt to resolve the dispute should proceed directly to step 2.

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

In accordance with relevant Codex Guidelines[6], the following information should be shared between competent authorities of the importing and exporting country to allow comparison of the results and procedures of the laboratory of the exporting country and its counterpart in the importing country. The relevant information covers:

- validation status of the methods of analysis used (including method specific sampling and preparation procedures),
- raw data (including spectral data, calculations, chemical standards used are assessed and are in order),
- results of repeat analysis,
- internal quality assurance/control (assessment of control charts, sequence of analysis, blank data, recovery data, uncertainty data, use of appropriate reference standards and materials),
- performance in relevant proficiency testing or collaborative studies and
- official accreditation status of the laboratories.

Each competent authority reviews its initial assessment on the basis of the additional information received from the other in order to recognise the validity of the results of one of the two laboratories (agreement on conformity or agreement on non conformity).

In this way, the dispute is resolved without further analysis or sampling.

If no agreement is reached, resolution of the dispute may be sought using the next step (where reserve

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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;

[5] Reference by Food Hygiene Committee

[6] See ANNEX to GUIDELINES FOR THE EXCHANGE OF INFORMATION BETWEEN COUNTRIES ON REJECTIONS OF IMPORTED FOOD (CAC/GL 25-1997): "Where imported food has been rejected on the basis of sampling and/or analysis in the importing country, details should be made available on request as to sampling and analytical methods and test results and the identity of the testing laboratory."

samples are available).

3.3. – STEP 3: NEW ANALYSES ARE CARRIED OUT

Prerequisites

If it is established that sample integrity has not been compromised in transit, there is an agreement on:

1. the sharing/swapping of the reserve samples,
2. the methods of analysis,
3. the laboratories involved: each laboratory may undertake new analyses or one laboratory in the presence of a representative of the other; or a third laboratory may be selected by consensus of exporting and importing country, or, failing that, by the competent authority of the importing country; and
4. the use of the new analytical results: either the initial results are discarded and the settlement of the dispute is determined by the comparison of the new results obtained; or the new results are used to confirm the validity of one of the two results obtained initially.

Available approaches

One (or more) may be selected.

A) – SEARCH FOR LABORATORY BIAS

It may be agreed to check for laboratory bias, by testing common samples[7]. Performances are compared by testing a common sample with a known analyte content, preferably certified reference material. The original results are then corrected according to the bias found. If the results are in agreement, within the reproducibility limit, the dispute is settled.

B) – IDENTIFICATION OF A SAMPLING PROBLEM

The two laboratories may swap their reserve samples. If both laboratories confirm the original results received by the other one, a sampling problem is identified.

C) – ANALYSES OF RESERVE SAMPLES

The new analyses are performed on shared reserve samples. Either:

1. analyses are performed in one laboratory in the presence of a representative of the other laboratory. The new results are used to assess conformity.
2. the two laboratories carry 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.

3.4 – STEP 4: New samples taken from the consignment are analysed

The consignment is located in the importing country. At this stage, the initial test results are no longer taken into account. The modalities of sampling and analysis are decided by consensus.

At the request of the competent authority of the exporting country, a new sampling of the consignment is carried out and new analyses are performed in a laboratory selected by consensus or, failing that, by the competent authority of the importing country.

or

(Proposal of the European Community)

[3. OCCURRENCE OF A DISPUTE

A dispute within the meaning of these guidelines arises when the difference between the results obtained in the two laboratories is larger than the sum of their two expanded measurement uncertainties, and one of the two countries claims the non-compliance.

It would be expected that the expanded measurement uncertainties reported by the laboratories will not substantially exceed two times the value of the estimated reproducibility standard deviation (S_R) at the

[7] To investigate analytical differences (biases) between laboratories, the laboratories need to test samples with known analyte concentrations (usually duplicate split samples). It is not necessary to test or retest samples from the original consignment of product under dispute: this would only be required if a reassessment were needed. To provide a reasonable estimate of bias, several (split) samples should be analysed, one duplicate of each sample at each laboratory. The appropriate number of samples should be used for the estimate of the bias to be reliable.

concentration of interest if the laboratory is in “analytical control”.

4. The analytical results are compared taking into account measurement uncertainty

By providing the necessary documents, the laboratories involved demonstrate that they are accredited for the analyses concerned, and hence meet the prerequisites outlined above.

In accordance with relevant Codex Guidelines[8], the following information should be shared between Competent Authorities of the importing and exporting country to allow comparison of the results and procedures of the laboratory of the exporting country and its counterpart in the importing country. The relevant information covers:

- validation status of the methods of analysis used and a method description (including method specific sampling and preparation procedures),
- raw data (including spectral data, calculations, chemical standards used)
- results of replicate analyses,
- internal quality assurance/control procedures (control charts, sequence of analysis, blank data, recovery data, recovery correction, uncertainty data, use of appropriate reference standards and materials),
- official accreditation status of the laboratories and
- performance in relevant proficiency testing schemes.

Each competent authority reviews its initial assessment on the basis of the additional information received from the other in order to recognise the validity of the results of each of the laboratories. If the results from each laboratory are accepted, then the importing country will use its own result to assess the compliance.

If the result from one laboratory is agreed not to be acceptable, then the result from that laboratory is discarded and the consignment is either accepted/rejected on the basis of the remaining result.

In this way, the dispute is resolved without further analysis or sampling.

If no agreement is reached, the dispute may be resolved as described below.

5. FURTHER ANALYSES ARE CARRIED OUT

Prerequisites

If it is established that sample integrity has not been compromised in transit, there is an agreement on:

1. the sharing/swapping of any reserve samples,
2. the methods of analysis to be used by each laboratory,
3. whether there is any laboratory bias (i.e. it may be agreed to check for laboratory bias by testing common samples[9].

RESOLUTION BY EVALUATION OF THE LABORATORY BIAS

Results from each laboratory are compared by testing a common sample with a known analyte content, preferably certified reference material. The original results are then corrected if a bias has been found. If the results, taking into account the measurement uncertainty, show that the same decision on compliance by both laboratories of the importing and exporting countries is found, then the dispute is resolved.

ANALYSES OF RESERVE SAMPLES

If necessary further analyses may be carried out on:

- any reserve samples taken by the exporting country but then analysed by a further designated laboratory in the importing country,
- the split sample taken on importation but analysed by a second designated laboratory in the importing country or

[8] See ANNEX to GUIDELINES FOR THE EXCHANGE OF INFORMATION BETWEEN COUNTRIES ON REJECTIONS OF IMPORTED FOOD (CAC/GL 25-1997): "Where imported food has been rejected on the basis of sampling and/or analysis in the importing country, details should be made available on request as to sampling and analytical methods and test results and the identity of the testing laboratory."

[9] To investigate analytical differences (biases) between laboratories, the laboratories need to test samples with known analyte concentrations (usually duplicate split samples). It is not necessary to test or retest samples from the original consignment of product under dispute: this would only be required if a reassessment were needed. To provide a reasonable estimate of bias, several (split) samples should be analysed, one duplicate of each sample at each laboratory. The appropriate number of samples should be used for the estimate of the bias to be reliable.

. • the second sample taken on importation but analysed by a second designated laboratory in the importing country.

If any of the above analyses show the consignment to be unsatisfactory, the consignment is considered to be out of compliance with the Codex specification.

NEW SAMPLES TAKEN FROM THE CONSIGNMENT IF IT IS STILL AVAILABLE

The consignment is located in the importing country. At this stage, the initial test results are no longer taken into account. The modalities of sampling and analysis are decided by consensus.

It might be agreed upon to carry out sampling and analysis in the presence of representatives of both parties involved.

At the request of the competent authority of the exporting country, a new sampling of the consignment is carried out and new analyses are performed in a laboratory selected by consensus or, failing that, by the competent authority of the importing country.

The results of this analysis are used to assess conformity. The dispute is settled.**]**

or
(Proposal of New Zealand)

[3.1. - STEP 1: THE ANALYTICAL RESULTS ARE COMPARED

If, and only if, the laboratories in the importing and exporting country have tested duplicates of the same samples, the following procedure can be used:

When the average difference between the test results is within the modified reproducibility limit, modified to account for the use of more than a single sample, as defined in the ANNEX, the importing country's test results should be used to assess conformity, taking into account measurement uncertainty.

The estimates of reproducibility to be used should be as follows, in order of precedence. For the importing country's results:

Those derived from published estimates of precision parameters for the method specified in the compliance test, or if these do not exist

Those derived from published estimates of precision parameters for the method actually used by the importing country, or if these do not exist

Estimates based on Horwitz or other appropriate models.

For the exporting country's results:

Those derived from published estimates of precision parameters for the method actually used by the exporting country, or if these do not exist.

Estimates based on Horwitz or other appropriate models.

The ANNEX suggests a simple procedure, based on the Horwitz model, to implement this criterion and resolve the dispute.

If the average result differs by more than the reproducibility limit, or if the Horwitz or other recognised models do not apply, the attempt to resolve the dispute should proceed to step 2.

If the two laboratories test different samples and an estimate of sampling uncertainty, representing the inhomogeneity of the analyte within the lot concerned, is available then a modified limit, akin to the reproducibility, incorporating both measurement uncertainty and that due to sampling variation within the lot concerned, is calculated and the procedures outlined in section (a) are applied using this limit.
If the average results differ by more than the modified reproducibility limit, or if the Horwitz or other models do not apply, the attempt to resolve the dispute should proceed to Step 2.

3.2. - STEP 2: NEW ANALYSES ARE CARRIED OUT (previously 3.3 – Step 3)

If it is established that sample integrity has not been compromised in storage or transit, and there is an agreement on:

The sharing/swapping of the reserve samples,

The methods of analysis,

then new analyses can be carried out.

Reference samples, could also be used at this step, as described in 3.4.

If, as required in the Prerequisites, the importing laboratory has retained reserve samples of the samples on which the finding of alleged non-conformity was based, then one duplicate from each sample is sent to a laboratory nominated by the exporting country for testing. The test results are compared using the measures outlined in Step 1 (a) of this procedure. If the averages of the test results from the two laboratories differ by less than the reproducibility limit appropriate for such an average, (see Annex), the importing country's assessment of the lot shall stand, and the dispute is thus resolved.

Otherwise, if duplicates of these split samples remain, then the measures outlined in Step 4 of this procedure, using arbitration by a third laboratory, can be applied.

However, if duplicates of the original, allegedly non-conforming samples do not exist, then the following measures, involving the exchange and testing of reserve samples, can be undertaken, where conditions (1) and (2) above have been met, subject to agreement on the following:

The laboratories involved: Each laboratory may undertake new analyses or one laboratory may test the samples in the presence of a representative of the other; or a third laboratory may be selected by consensus of the exporting and importing country. Failing agreement on the choice of laboratory the competent authority of the importing country can select a laboratory.

The use of the new analytical results: Either the initial results will be discarded and the settlement of the dispute determined by the comparison of the new results obtained; or the new results used to confirm the validity of one of the two results obtained initially.

3.3. - STEP 3: THE RESULTS AND PROCEDURES OF THE LABORATORY OF THE EXPORTING COUNTRY AND ITS COUNTERPART IN THE IMPORTING COUNTRY ARE COMPARED (previously 3.2 – Step 2)

In accordance with relevant Codex Guidelines[10], the following information should be shared between competent authorities of the importing and exporting countries to allow comparison of the results and procedures of the laboratory of the exporting country and its counterpart in the importing country. The relevant information covers:

- validation status of the methods of analysis used (including method specific sampling and preparation procedures),
- raw data (including spectral data, calculations, chemical standards used are assessed and are in order),
- results of repeat analyses,
- internal quality assurance/control (assessment of control charts, sequence of analysis, blank data, recovery data, uncertainty data, use of appropriate reference standards and materials),
- performance in relevant proficiency testing or collaborative studies, and
- official accreditation status of the laboratories.

Each competent authority reviews its initial assessment on the basis of the information received from the other in order to recognise the validity of the results of one of the two laboratories and agree on conformity or non conformity.

3.4. - STEP 4: ANALYSIS OF REMAINING DUPLICATES OF RESERVE SAMPLES

Where third duplicates of the samples on which the finding of non-conformity was based are available, these should be analysed by a suitably qualified laboratory agreed on by the two countries, and a final assessment of conformity based on the results from this laboratory. The original results and the results from the second duplicate tested under Step 2 are discarded. If possible this laboratory should be independent of the two laboratories whose results were compared in step 2. It is highly desirable that a Certified 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

[10] See ANNEX to GUIDELINES FOR THE EXCHANGE OF INFORMATION BETWEEN COUNTRIES ON REJECTIONS OF IMPORTED FOOD (CAC/GL 25-1997): "Where imported food has been rejected on the basis of sampling and/or analysis in the importing country, details should be made available on request as to sampling and analytical methods and test results and the identity of the testing laboratory."

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

If a third sample is not available, resolution of the dispute may be sought using Step 5.

3.5. - STEP 5: New samples taken from the consignment are analysed [previously 3.4 – Step 4]
It is assumed that the consignment is located in the importing country. At this stage, the initial test results are no longer taken into account. The modalities of sampling and analysis are decided by consensus.

At the request of the competent authority of the exporting country, a new sampling of the consignment is carried out and new analyses are performed in a laboratory selected by consensus or, failing that, by a laboratory selected by the competent authority of the importing country.

ANNEX
Modified reproducibility limit for the mean of several samples when duplicates of each of the samples are tested by each laboratory:

This is given by

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

where

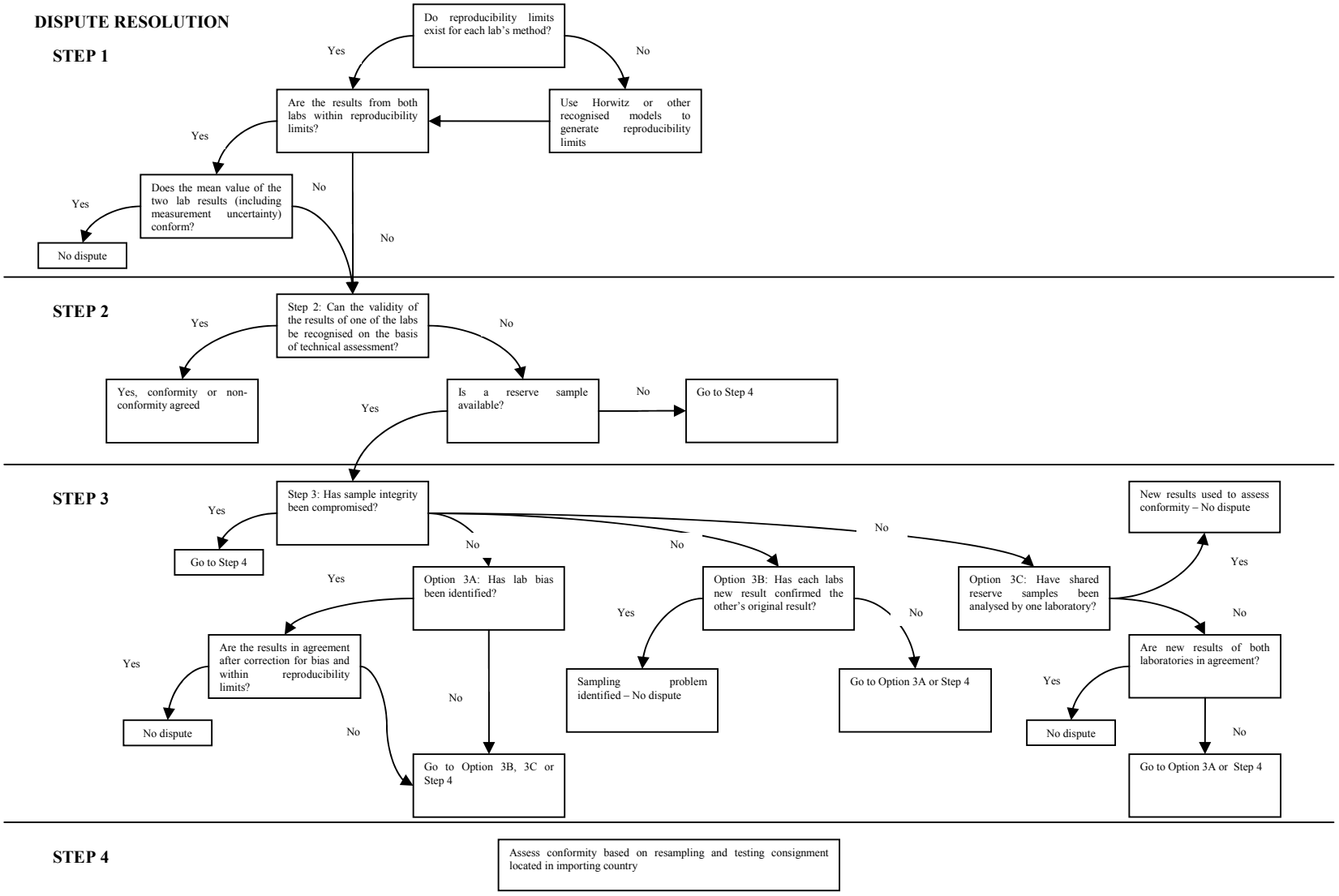
n is the number of samples over which the mean is taken
s_R is the reproducibility standard deviation
s_r is the repeatability standard deviation.

If there is no published repeatability for the method, or if the Horwitz model is used, s_r could be taken as approximately $0.5 s_R$.

<Figures 1 and 2 and Table 1 (Alinorm 07/30/23, Appendix IV) should be included.>

ANNEX 1

FLOWCHART



Definition of a maximum acceptable difference Δ_{max}

Let define the average contents of the sample *T* and the relative difference between results Δ% as:

$$T = \frac{Y_1 + Y_2}{2}$$

$$\Delta\% = \frac{|Y_1 - Y_2|}{T} \times 100$$

The acceptance condition is that the difference between both results is below reproducibility limit defined in ISO 5725 from the reproducibility standard deviation s_R:

$$|Y_1 - Y_2| \leq 2.83 s_R$$

If there is no published reproducibility, it is possible to use the model of Horwitz to calculate the limit of reproducibility as:

$$s_R = 0.02 \times T^{0.8495}$$

Then it comes:

$$|Y_1 - Y_2| \leq 0.0566 \times T^{0.8495}$$

Thus, the maximal acceptable difference (relative) is:

$$\Delta_{max} \leq \frac{0.0566 \times T^{0.8495}}{T} \times 100$$

Figure 1 illustrates, as an abacus, this decision criterion. When dealing with concentration around 1 ppm, the relative difference between results must be below 45%. This value seems rather high but, for instance, it is often consistent with the toxicological meaning of a contaminant. When available or recognized other models than Horwitz's could be used (see Table **1**).

Measurement uncertainty of the mean

Let define u_1 and u_2 as the measurement uncertainty of each individual test results Y_1 and Y_2 respectively, then the measurement uncertainty of the mean is:

$$u_{mean} = \sqrt{\frac{u_1^2 + u_2^2}{4}}$$

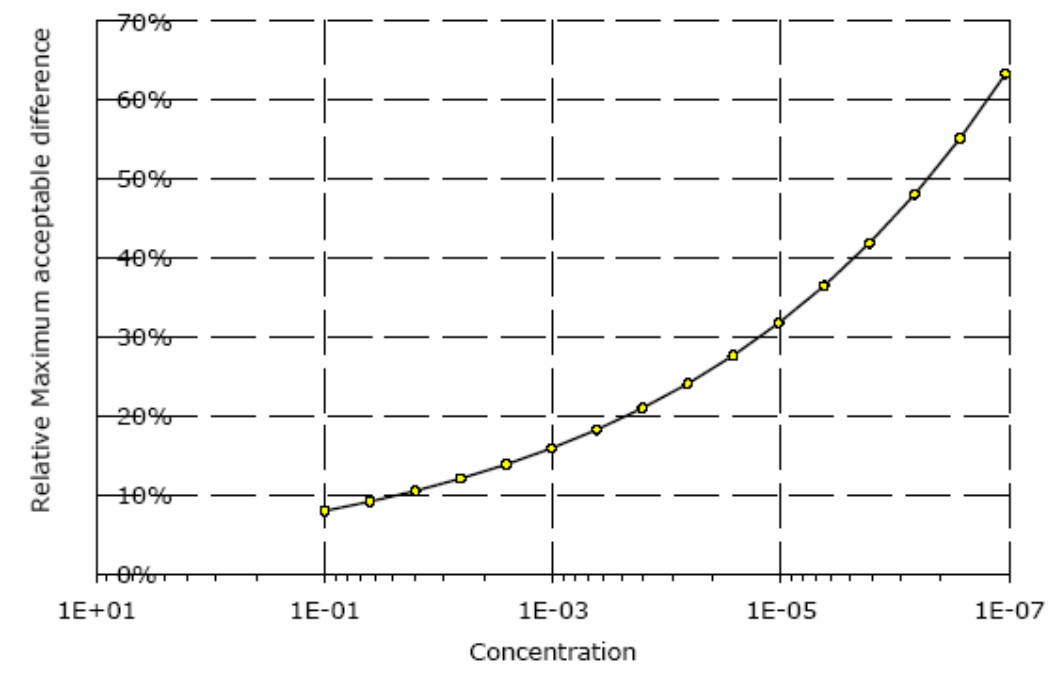


Figure 1. Relative Maximum acceptable difference based on Horwitz's model

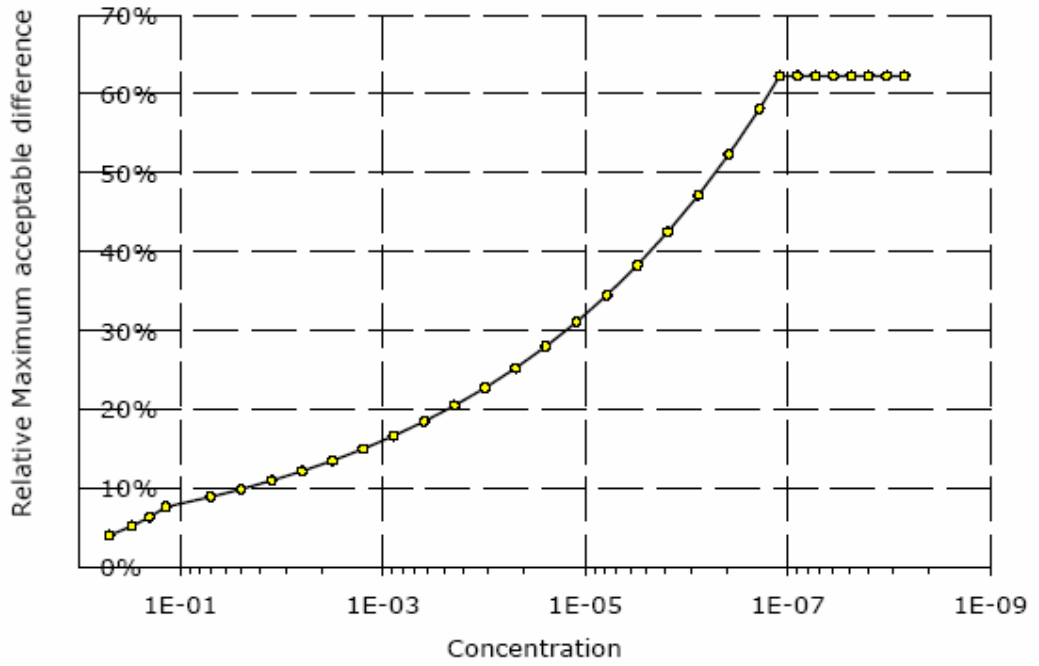


Figure 2. Relative Maximum acceptable difference based on Thompson's model

Table 1. Published recognized models

Name	Range (dimensionless)	Equation of s_R	Equation for Δ_{max} (%)	Figure
Horwitz [1]	10^{-1} to **1.2** 10^{-7}	$s_R = 0.02 \times T^{0.8495}$	$\Delta_{max} \leq \frac{5.66 \times T^{0.8495}}{T}$	**1**
Thompson [2]	$> 1.38\ 10^{-1}$	$s_R = 0.01 \times T^{0.5}$	$\Delta_{max} \leq \frac{2.83 \times T^{0.5}}{T}$	**2**
	$1.38\ 10^{-1}$ to $1.2.10^{-7}$	$s_R = 0.02 \times T^{0.8495}$	$\Delta_{max} \leq \frac{5.66 \times T^{0.8495}}{T}$	
	$< 1.2.10^{-7}$	$s_R = 0.22 \times T$	62.26%	

References

[1] Horwitz W. (1980) Quality Assurance in the Analysis of Foods for Trace Constituents, *J of the AOAC* 63:6, 1344-1354

[2] Thompson M. (2000) Recent trends in inter-laboratory precision at ppb and sub-ppb concentrations in relation to fitness for purpose criteria in proficiency testing, *Analyst* 125, 385-386

ANNEX 3

BRAZIL: Revised Flowchart