CODEX ALIMENTARIUS COMMISSION

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World Health Organization

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JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING
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DRAFT REVISED GUIDELINES ON MEASUREMENT UNCERTAINTY
(at Step 6 of the Procedure)
(Comments of Argentina, Brazil, Canada, Cuba, the European Union, Japan, New Zealand and the United States of America)

ARGENTINA

Argentina submits these comments:

In **7 Values of Measurement Uncertainty Estimations** we believe the first paragraph is unclear (both the English version and the Spanish translation), so we propose to replace the original wording:

"Stipulating information on the anticipated values of measurement uncertainty estimations is frequently not supported by analysts. The users of analytical data and the customers of the laboratories producing such data frequently ask for such information regarding the level of uncertainty that may be expected for test results. They have concerns that some laboratories underestimate the size of their uncertainties and so report unrealistically small uncertainties to their customers."

with the following:

"Although it is acceptable for laboratories to adopt, for their tests, values of uncertainty established by technically recognized organizations, it is recommended that each laboratory conduct its own estimation in accordance with the suggestions in paragraphs 5 and 6 of this document, so the reported uncertainty data will be representative of the performance of the method validated or verified within the laboratory, preventing reporting unrealistically small uncertainties to customers".

We believe the references should be included below the table in 7 and 8.2.

In the Spanish version, "8.2 Recuperación" should be moved below the explanatory diagram, and the words "Índice de recuperación" should be replaced with **"recuperación porcentual o porcentaje de recuperación".**

BRAZIL

Brazil would like to propose eliminating the structure based on questions and answers and using the following titles for the items: Brazil thanks the United Kingdom for the preparation of this document and proposes new wording to:

General amendment of the document:

Brazil would like to propose eliminating the structure based on questions and answers and using the following titles for the items:

In the item 1 substituting [1. What is Measurement Uncertainty?] by 1. The meaning of Measurement Uncertainty

In the item 2 substituting [2. Does the Measurement Uncertainty have to be Estimated in Codex?] by 2. Why the Measurement Uncertainty should be estimated and excluding the word [Yes,] starting the sentence "One of the requirements of….".

In the item 3 deleting [3. Does Measurement Uncertainty Arise From both Sampling and Analysis?] in addition, moving the text "Measurement uncertainty applies to the whole measurement process. However, this guidance only considers analytical measurement uncertainty." to the beginning to become it the first sentence of the document.

In the item 4 substituting [4. What is the Relationship between Measurement Uncertainty, the Analytical Result and the Method Used to Obtain the Result?] by 4. Relationship between Measurement Uncertainty, the Analytical Result and the Method Used to Obtain the Result

The other titles are appropriate.

In the item 1, 1st. paragraph: excluding the text [It is not always appreciated that analytical results are variable, and just how large that variability may be, particularly when low concentrations of a measurand (i.e. ppb levels) are being determined. As stated in the Guidelines,] and starts the paragraph – "Measurement uncertainty applies to the whole measurement process. However, this guidance only considers analytical measurement uncertainty."

Substituting ["most quantitative analytical results take the form of "a ± 2u" or "a ± U" where "a" is the best estimate of the true value of the concentration of the measurand (the analytical result) and "u" is the standard uncertainty to 68% level of confidence and "U" (equal to 2u) is the expanded uncertainty to 95% level of confidence. The range "a ± 2u" represents a 95% level of confidence in which the true value would be found. The value of "U" or "2u" is the value, which is normally used and reported by analysts, normally referred to as "measurement uncertainty" and may be estimated in a number of different ways.] by "Most quantitative analytical results take the form of "a ± ku or a ± U" where "a" is the best estimate of the true value of the concentration of the measurand (the analytical result), "k" a coverage factor and "u" is the standard uncertainty and "U" (equal to ku) is the expanded uncertainty. The range "a ± ku" (where k = 2) represents a 95% level of confidence where the true value would be found (in other cases can be increased as k = 3 for 99% level of confidence). The value of "U" or "ku" is the value which is normally used and reported by analysts and is hereafter referred to as "measurement uncertainty" and may be estimated in a number of different ways."

In the item 8.1 Measurement Uncertainty

Use the following title for diagram on page 72.

"Assessment of compliance with and Upper Limit"

CANADA

Canada is pleased to offer the following comments on the Draft Revised Guidelines on Measurement Uncertainty:

The English version of the document is lacking a diagram in section 8.1. The French version does contain the diagram which is especially useful for non-technical users of the document. Canada suggests that the diagram be included in both the English and French versions as a visual means in order to make the impact of uncertainty on decision making clearer.

This document, which was drafted at the last CCMAS meeting in March 2010, is acceptable to Canada as it merely outlines responsibilities of laboratories to estimate uncertainty using recognized, but not specified, methods as required to meet the principles of ISO 17025. It also demonstrates how uncertainty may be used in decision making but does not mandate how nor require countries to do so.

CUBA

Cuba has no comments on the **Draft Revised Guidelines on Measurement Uncertainty** at Step 6, as we agree with all of this document so far.

EUROPEAN UNION

> *Mixed Competence.*
> *Member States Vote.*

The European Union and its Member States (EUMS) are generally supporting the revised guidelines, but would however suggest the following **comments of an editorial nature** on the "*Explanatory Notes to the Codex Guidelines on Measurement Uncertainty*".

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1 What is Measurement Uncertainty?

…

In food analysis it is the (approximately) 95% ~~probability~~ **level of confidence** (i.e. 2u) which is used to calculate the expanded uncertainty. Other sectors may specify a different ~~probability~~ **level of confidence**.

…

8.1 Measurement Uncertainty

Situation I

The analytical result together with the measurement uncertainty exceeds the maximum level. The result indicates that the ~~measured~~ analyte ~~in the sampled lot~~ is above the specification.

Situation II

The analytical result exceeds the maximum level ~~by less than the measurement uncertainty~~ but with the lower endpoint of the measurement uncertainty less than the maximum level.

Situation III

The analytical result is less than the maximum level but with the upper endpoint of the measurement uncertainty being greater than the **maximum** level.

Situation IV

The analytical result bounded by the expanded measurement uncertainty is less than the maximum level.

This diagram demonstrates the importance of defining clear guidelines to allow unambiguous interpretation of analytical results with respect to their measurement uncertainties.

In addition, the EUMS would propose to re- insert the diagram at the end of Section 8.1. This diagram was present in Appendix IV of ALINORM 10/32/23 but was removed in the text appended to CL 2010/49-MAS.

JAPAN

Japan notices that the diagram of this section is deleted. If this deletion is not a mistake, some explanations concerning the diagram become unnecessary.

> *8.1 Measurement Uncertainty*
>
> It is important that measurement uncertainty is considered when deciding whether or not a sample meets the specification. This consideration may not apply when a direct health hazard is concerned. ~~The significance of this can be illustrated by an example shown in the diagram below, which shows the simplest case when decisions are made based on a single test sample.~~
>
> The example ~~shown here~~ is one where the test result is compared against the specification consisting of a maximum level.
>
> > *Situation I … Situation IV*
> >
> > ~~This diagram demonstrates the importance of defining clear guidelines to allow unambiguous interpretation of analytical results with respect to their measurement uncertainties.~~

NEW ZEALAND

General comments

New Zealand supports progressing the *Explanatory Notes to the Codex Guidelines on Measurement Uncertainty* towards finalisation, but we are concerned that at present some points are unhelpful, misleading or incorrect, and some important information is omitted. The faults defeat the purpose of providing helpful explanation, and instead create potential that the information may be misunderstood or misused, and may create barriers to trade, so we could not agree to progress the *Notes* without corrections.

However corrections are not straightforward because of incorrect material already incorporated in the main body of the *Guidelines*. New Zealand has proposed corrections to the *Notes* below, aiming to make the corrections without contradicting the *Guidelines* in order to avoid confusion.

In view of the faults in the *Guidelines* New Zealand would also agree to deferring work on the *Notes* and instead focusing on corrections to the main body of the *Guidelines.*

Our comments on specific text are as follows.

Section 1, What is measurement uncertainty?

Paragraph 1. The first sentence raises a number of questions but leaves them unanswered. For instance, is it referring to results from one sample? What is variability and how does it relate to the term "dispersion" used in the definitions? What is the significance of the size of variability? Without clarity on these points the sentence is not useful.

The quote from the *Guidelines* is not needed as it is merely repetition.

We suggest the paragraph should be deleted.

Paragraph 2. There is no need to state the coverage factor conventionally used in food analysis, nor to comment on other sectors. This wording should be deleted. Instead we suggest that since the conventional estimate of expanded uncertainty, *2u*, is used in other Codex documents, this should be mentioned along with a comment that a different coverage factor may be needed for other purposes, since the factor *k=2* may considerably underestimate the expanded uncertainty. It would also be useful to include a caution against the use of the Student's *t*-distribution to derive coverage factors. We suggest:

The conventional estimate of expanded uncertainty *U* (equal to *2u*) is used for some Codex purposes; this effectively assumes that the estimate *u* is exact. Where the uncertainty is estimated from limited data, a different coverage factor may be needed to achieve the appropriate confidence in the desired level of coverage.

However it should be noted that the appropriate factor for 95% coverage should *not* be derived from the 95th percentile of the Student's *t*-distribution. Such a factor, defined on the degrees of freedom, would provide only 50% confidence that 95% coverage is achieved.

Paragraph 3. This paragraph again explains measurement uncertainty in terms of variability rather than the term "dispersion" used in the definitions. In addition, the concept of anticipating a "true" result is a Bayesian approach that is radically different from and not compatible with the frequentist approach used elsewhere by Codex. Paragraph 3 should therefore be deleted.

Section 2, Does the measurement uncertainty have to be estimated in Codex?

In line 2, replace "must" with "should", since this is a guideline.

In lines 2 and 3, delete the words, "or when the uncertainty affects compliance to a specification limit, for example a Codex Standard". This is not one of the reasons for estimating measurement uncertainty, or making it available, either in ISO/IEC 17025 or the *Guidelines on Measurement Uncertainty*.

In the last sentence, the motive which might prompt a request for measurement uncertainty information should be mentioned. We suggest that the sentence could read:

As Codex is concerned with goods moving in international trade it would be anticipated that the request may be made, for instance to assist in the resolution of a dispute over analytical (test) results.

Section 3, Does measurement uncertainty arise from both sampling and analysis?

The heading implies that measurement uncertainty could cover sampling. This is not clear from the 2 Codex definitions, where measurement uncertainty is linked only to measurement. To avoid confusion the word "measurement" should be deleted from the heading.

The paragraph is also ambiguous, since it is not clear what is covered by the "whole" measurement process, or by analytical measurement uncertainty. It could be clarified by adding an explanation of what is meant by the whole measurement process, and replacing the second sentence with a sentence indicating that uncertainty also arises from sampling. It could read :

Measurement uncertainty applies to the whole measurement process, from receipt of samples through to issuance of analytical reports. Uncertainty also arises from sampling.

Section 4, What is the relationship between measurement uncertainty, the analytical result and the method used to obtain the result?

The first sentence is not relevant to the question and should be deleted.

The first clause of the second sentence is not correct as it stands, since the precision of the method certainly contributes to measurement uncertainty. The second clause is rather ambiguous. We suggest the second sentence should be replaced as follows:

The precision of a method of analysis contributes to measurement uncertainty. The precision values that are obtained in the validation and/or in quality control of a method may be used as an element of the estimation of the measurement uncertainty of a result, or in some situations may constitute the whole of the measurement uncertainty.

The last sentence omits the important point that reliable estimates of measurement uncertainty from specific laboratories can be used in place of precision data. We suggest the last sentence should be amended as follows:

In particular, they may be superseded by more specific estimates relating, for example, to a specific laboratory, if such estimates are sufficiently reliable. Also, additional factors such as uncertainty associated with bias, matrix effect, and competence of laboratory must be considered.

Section 5, Procedures for estimating measurement uncertainty

In paragraph 1, the discussion about scientific credibility and appropriateness of the estimation procedures is not useful because there is no measure of performance for a statement of measurement uncertainty. Furthermore, since Codex does not recommend a particular approach to estimation of measurement uncertainty, the word "recognised" (line 4) and the last sentence are not appropriate. Therefore the wording from "but it is important …" (line 2) to the end of the paragraph should be deleted.

Paragraph 4 states that method bias is not included in validation studies. We believe it is normally included, but on the other hand the imprecision in estimating bias (both method bias and laboratory bias) is not included. The third indent should therefore be replaced by "Imprecision in estimating method or laboratory bias".

Section 6, Considerations when estimating measurement uncertainty within the context of Codex

This section does not need to be stated for Codex purposes.

Section 7, Values of measurement uncertainty estimations

The purpose of this section is not clear, and we suggest it could be deleted.

If it is retained, the source and validity of the indicative values should be made available, and recommended actions to be taken if a laboratory's estimate of measurement uncertainty does not conform to the typical values should be included.

SECTION 8.1, MEASUREMENT UNCERTAINTY

The *Notes* should explain that uncertainty of sampling is important, and that in many cases measurement uncertainty is not significant in comparison and does not need to be allowed for. We suggest wording as follows, following the second sentence:

In many cases uncertainty of sampling is as large as or larger than measurement uncertainty. Uncertainty of sampling is often the overriding factor in conformity assessment procedures. Sampling procedures in the *General Guidelines on Sampling* are designed to take account of uncertainty of sampling.

The decision procedure described in this section is adopted by Codex only for certain specific situations, and has serious limitations. Notes should be included to avoid the impression that the procedure is generally applicable. Sentence 3 should be followed by a note that the decision procedure is not one that is generally recommended by Codex, as follows:

Note that this decision procedure has not been recommended by Codex for general use, but it could be appropriate in certain specific situations.

In paragraph 2, the explanation of the example should note that uncertainty of sampling is being ignored. We suggest wording as follows:

Uncertainty of sampling is ignored; so in many situations this example is not applicable.

In *Situation I*, the second sentence is misleading and should be deleted.

The last paragraph emphasises that national governments should define guidelines for interpretation of the situations illustrated. However since the situations are open to different interpretations, this will lead to differing practices and to disputes. The paragraph should be deleted, or alternatively clear guidelines should be included.

Section 9, Useful references

The references that are not endorsed by Codex should not be included in the *Notes*. References should be endorsed only when they have been shown to be appropriate, scientifically credible, and available.

UNITED STATES OF AMERICA

The United States is pleased to submit the following comments on the Codex Proposed Draft Revised Guidelines on Measurement Uncertainty (CAC/GL 54-2004) circulated at Step 6 in **CL 2010/49-MAS** for comments and consideration by the 32nd Session of the Committee on Methods of Analysis and Sampling (CCMAS) (Budapest, Hungary, 7-11 March 2011).

This document will provide valuable explanatory information related to the Codex Guidelines on Measurement Uncertainty (CAC/GL 54-2004), and the United Kingdom is to be commended for its efforts in drafting and leading the discussion of this document.

(i)   General Comments

The United States offers the following minor editorial changes that are intended to provide clarity to the document and should not alter the substance or the meaning of its content.

(ii)   Specific Comments

Section 2: Does the Measurement Uncertainty have to be Estimated in Codex?

The following changes are recommended:

Yes, one of the requirements of the ISO/IEC 17025:2005 Standard that Codex has adopted by reference is that the measurement uncertainty of a result must be estimated and then made available if requested or when the uncertainty affects compliance to a specification limit, for example a Codex Standard~~. (t~~**The** Codex Alimentarius Commission has developed Guidelines~~,~~ (CAC/GL 27-1997) ~~which~~ **that** require laboratories involved in the import/export of foods to comply with general criteria in ISO/IEC 17025~~)~~. As Codex is concerned with goods moving in international trade it would be anticipated that the request **for measurement uncertainty estimates** will be made.

Section 4: What is the Relationship between Measurement Uncertainty, the Analytical Result and the Method Used to Obtain the Result?

The following changes are recommended in the first sentence:

~~It is the~~ **The** uncertainty of test results ~~which~~ is one of the factors when judging compliance with standards.

Section 5: Procedures for Estimating Measurement Uncertainty

The following changes are recommended for the third sentence of the first paragraph:

 No one approach may be said to be better than any other provided the procedure used is appropriate and credible**;** ~~i.e.~~ there is no "hierarchy" of the recognised procedures.

Also, in the first sentence of the fifth paragraph the following change is recommended:

For methods operating within their defined scope**s**,…

Furthermore, the following change is recommended in the last sentence of the sixth paragraph:

It is anticipated that procedures based on results obtained from participation in proficiency testing ~~**schemes**~~ **programs**, as an example, will be developed.

Section 6: Considerations when Estimating Measurement Uncertainty within the Context of Codex

In the second paragraph, the following changes are suggested:

When deciding on which procedure is to be used when estimating measurement uncertainty within the Codex context it is important to recognise ~~**which**~~ **that** Codex has adopted a number of formal quality assurance measures **that** have to be implemented by control laboratories. In particular, such laboratories should:

•   be in compliance with an Internationally recognised Standard (now with ISO/IEC 17025:2005 Standard); **(**such compliance is aided by the use of internal quality control procedures**)**, participate in proficiency ~~**schemes**~~ **testing programs**, and

Section 7: Values of Measurement Uncertainty Estimations

The following changes are recommended:

Change the title to read as follows: **Values of Measurement Uncertainty Estimations**

~~**Estimations**~~ **Estimates**

This will also cause the following change in the first sentence:

Stipulating information on the anticipated values of measurement uncertainty ~~estimations~~ **estimates** is frequently not supported by analysts.

In the second paragraph of the section, the following changes are proposed:

For chemical analyses, using the values of *sR* from collaborative trials, it would ~~**not**~~ be ~~**un**~~reasonable to anticipate that the (expanded) uncertainties reported by laboratories would be ~~**of the**~~ **approximately the** following ~~**orders**~~:

In the third paragraph of the section, the following changes are proposed:

It would be expected that the reported measurement uncertainties by ~~**all laboratories**~~ **any laboratory** would not significantly exceed the value estimated from the sR at the concentration of interest if the laboratory is in "analytical control". Very experienced laboratories carrying out any particular analysis on a regular basis would be expected to obtain **uncertainty** values less that the values given above.

Section 8: Relationship between analytical results, measurement uncertainty, and recovery factors

The sentence in this section should read:

This section attempts to explain the significance of analytical results and their associated measurement ~~**uncertainty**~~ **uncertainties** and ~~**recovery**~~ **recoveries**.

8.1 Measurement Uncertainty

The following changes are recommended for the third sentence of the first paragraph: The significance of this can be illustrated by an example ~~**shown in the diagram below**~~, which shows the simplest case when decisions are made based on a single test sample.

Also, in the second paragraph, the sentence should read:

The example ~~shown~~ **given** here is one where the test result is compared against the specification consisting of a maximum level.

Situation I

The following change is recommended for the first sentence of this section:

The analytical result ~~together with~~ **minus** the measurement uncertainty exceeds the maximum level.

Situation II

The following change is recommended for this section:

The analytical result exceeds the maximum level by less than the measurement uncertainty ~~but~~ (with the lower endpoint of the measurement uncertainty less than the maximum level).

Situation III

The following change is recommended for this section:

The analytical result is less than the maximum level ~~but with the upper endpoint of~~ the result plus the measurement uncertainty ~~being~~ greater than the **maximum** level.

8.2 Recovery

The following change is recommended for the second paragraph of this section:

Analytical results should be expressed on a **recovery-**corrected basis where appropriate and relevant, and when corrected ~~it has~~ **they have** to be stated **as such**.