codex alimentarius commission

FOOD AND AGRICULTURE ORGANIZATION OF THE UNITED NATIONS

WORLD HEALTH ORGANIZATION

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Agenda Item 7 **CX/MAS 04/8**

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING
Twenty-fifth Session
Budapest, Hungary, 8 – 12 March 2004

REVIEW OF THE *ANALYTICAL TERMINOLOGY FOR CODEX USE* IN THE PROCEDURAL MANUAL

Government Comments (Cuba, France, Iran, United States of America)

CUBA (Spanish version)

Dando respuesta a la CL 2003/43-MAS le informamos que la República de Cuba está de acuerdo con la terminología planteada en el documento sin modificación alguna.

CUBA (English version)

In reply to CL 2//3/43 –MAS, we inform you that the Republic of Cuba agrees with the terminology presented in the document without any modification.

FRANCE (French version)

Les autorités françaises pensent que, d'une façon générale, il convient de citer les sources des définitions, lorsque tout ou partie de la définition d'un terme est la reprise intégrale d'une Norme (ex: ISO 3534-1 pour justesse, fidélité, répétabilité, reproductibilité).

Dans les observations qui suivent les autorités françaises se sont directement inspirées des travaux menés par l'Association française de normalisation (AFNOR), dont les résultats ont été publiés dans le document intitulé FD V01-000 - *Analyse des produits agricoles et alimentaires- Terminologie (1999).*

Les observations sont présentées dans l'ordre du texte Codex en vigueur et les passages à modifier sont surlignés :

Résultat:

(…)

Remarques: (…)

Une expression complète du résultat d'une mesure inclue des informations sur l'incertitude de la mesure **(ajouter)** ***et sur l'unité.*** (…)

Etude *inter-laboratoires* ***(remplacer 'étude » par « essai »,terme exact ici, et en accord avec le Guide ISO 43-1) :***

(…)

Etude *de la performance des méthodes* ***(remplacer ici et ultérieurement « étude « par « essai »):***

Remarques:

(…) Habituellement, l'analyste n'a pas connaissance de la composition véritable des échantillons pour essai, mais connaît la matrice. Le nombre de laboratoires, le nombre d'échantillons pour essai, le nombre de déterminations et autres détails sont spécifiés dans le protocole de l'étude. **Une partie de ce protocole contient la procédure précisant les directives écrites pour réaliser l'analyse (*phrase peu claire, à remplacer par « Le document d'organisation de l'essai décrit en particulier les détails de la méthode d'analyse que devront utiliser les laboratoires participant à l'essai »*).**

La principale caractéristique de ce type d'étude est la nécessité d'adopter exactement le même protocole écrit et la même méthode ~~expérimentale~~**d'analyse**

Plusieurs méthodes peuvent être comparées à l'aide des mêmes matériaux pour essai. **Si tous les laboratoires utilisent la même série de directives pour chaque méthode*(phrase peu claire, à remplacer par « Si tous les laboratoires appliquent scrupuleusement chacune des méthodes »)*** (…).

Étude de performance des laboratoires:

(…)

Remarques:

On peut utiliser les essais d'aptitude des laboratoires pour appuyer l'accréditation des laboratoires ou en contrôler les performances. Si une étude est réalisée ***(ajouter) en appui*** à une organisation ayant un certain pouvoir de gestion sur les laboratoires participant - administration, accréditation, réglementation ou sous-traitance - la méthode peut être spécifiée ou le choix limité à une liste de méthodes approuvées ou équivalentes. En pareil cas, ~~**un échantillon pour essai unique**~~ ***(faux, remplacer par « un seul essai »)*** est insuffisant pour juger la performance. (…)

Étude de certification de matériau: Étude inter-laboratoires qui attribue une valeur de référence ***("valeur (ajouter) conventionnellement vraie")*** à une quantité (concentration ou propriété) d'un matériau pour essai, habituellement avec une d'incertitude connue. (…)

TERMES POUVANT ETRE UTILISES DANS LA DEMARCHE-CRITERES

Limite de détection

(introduire d'abord une définition générale, telle que dans la publication AFNOR FD V 01-000, « Plus petite concentration ou teneur de l'analyte pouvant être détectée, mais non quantifiée, dans les conditions expérimentales décrites de la méthode »)

La limite de détection est définie conventionnellement comme échantillon à blanc + 3ó, où ó est l'écart type de l'indice de valeur de l'échantillon à blanc (définition UICPA). Cependant, une définition alternative qui répond à la plupart des objections à l'approche ci-dessus (à savoir la grande variabilité à la limite de mesure ne peut pas être résolue) est de se baser sur l'écart-type arrondi de la reproductibilité lorsqu'il n'est plus sous contrôle (3óR= 100%; 3óR= 33%, arrondi à 50% du fait de la grande variabilité). Une telle valeur est directement liée à l'analyte et au système de mesure est n'est pas basé sur ~~le système de mesure local~~ l'appareillage de mesure utilisé.

Limite de détermination (utiliser plutôt le terme « quantification », qui permet une plus claire distinction de la notion de limite de détection)

(ici aussi, introduire une définition générale, telle que celle du FD V 01-000 « Plus petite concentration ou teneur de l'analyte pouvant être quantifiée avec une incertitude acceptable, dans les conditions expérimentales décrites de la méthode) (…)

FRANCE (English version)

The French authorities think that in general, the sources of the definitions should be mentioned, when a Standard has been used for the definition or part of it (example: ISO 3534-1 for trueness, precision, repeatability, reproducibility)

The following comments of the French authorities are directly based on the work of the French Association for Standardization (AFNOR) the results of which have been published is document FD V01-000 – *Analysis of agricultural and food products – Terminology* (1999).

The comments are presented in the order of the current Codex text and the sections to be amended are highlighted:

Result:

(…)

Notes: (...)

A complete statement of the result of a measurement includes information about the uncertainty of measurement **(add) and unit**.

Interlaboratory Study: replace "Study" with "Test", as this is the exact term, in conformity with ISO Guide 43-1

(...)

Method-Performance Study (replace "Study" with "Test" here and hereafter)

Notes:

(...) Usually the analyst is not aware of the actual composition of the test samples but is aware of the matrix.

The number of laboratories, number of test samples, number of determinations, and other details of the study are specified in the study protocol. Part of the study protocol is the procedure which provides the written directions for performing the analysis. **(this is not clear, to be replaced with "The document concerning the organisation of the test describes in particular the details of the method of analysis that the laboratories participating in the test should use.")**

The main distinguishing feature of this type of study is the necessity to follow the same written protocol and ~~test~~ method **of analysis** exactly.

Several methods may be compared using the same test materials. If all laboratories use the same set of directions for each method **(this is not clear, to be replaced with "If all laboratories scrupulously apply each of the methods")** (...)

Laboratory-Performance (Proficiency) Study

Notes:

Laboratory-performance studies can be used to support accreditation of laboratories or to audit performance. If a study is conducted **(add) in support to** an organization with some type of management control over the participating laboratories—organizational, accreditation, regulatory, or contractual—the method may be specified or the selection may be limited to a list of approved or equivalent methods. In such situations, ~~a single test sample~~ **(incorrect, replace with a "single test")** is insufficient to judge performance. (...)

Material-Certification Study: An interlaboratory study that assigns a reference value ("true value") **(add "conventionally true value")** to a quantity (concentration or property) in the test material, usually with a stated uncertainty. (...)

TERMS TO BE USED IN THE CRITERIA APPROACH

Detection Limit

(First introduce a general definition, such as that in the publication AFNOR FD V 01-000 "Lower concentration or level of the analyte that may be detected, but not quantified, in the test conditions described for the method")

The detection limit is conventionally defined as field blank + 3σ, where σ is the standard deviation of the field blank value signal (IUPAC definition).

However, an alternative definition which overcomes most of the objections to the above approach (i.e. the high variability at the limit of measurement can never be overcome) is to base it on the rounded value of the reproducibility relative standard deviation when it goes out of control (where 3 σ_R = 100%; σ_R = 33%, rounded to 50% because of the high variability). Such a value is directly related to the analyte and to the measurement system and is not based on the ~~local measurement system~~ **measurement equipment** used.

Determination limit (use rather "quantification" that allows a clearer distinction of the notion of limit of detection)

(here also a general definition should be introduced, such as that of FD V 01-000 "Lower concentration or level of the analyte that may be quantified with an acceptable uncertainty, in the experimental conditions described in the method)

(...)

IRAN

Iran wishes to make the following comments of a largely editorial nature regarding the Review of the Analytical Terminology for Codex Use in the Procedural Manual:

General comment

·Modifying the text in a way that the sentences become clearer is recommended.

Specific comments

·Page 2, in paragraph 5 line 4, "the absence" should read " Omission" . ·Page 2, in paragraph 7 line 1: The term accuracy as concept should read as follows: "The closeness of agreement between obtained value and conventional true value or accepted reference value".

·Page 2, in paragraph 9 line 1: The term accuracy as statistic should read as follows: "The closeness of agreement between obtained value and conventional true value or accepted reference value".

·Page 2, in paragraph 13 line 1, "Trueness has been referred to as accuracy of the mean " As stating the mean without standard deviation has a limited value and sometimes could be misleading, this should be replaced by the following new sentence: "Trueness has been referred to as "mean of accuracy + standard deviation".

·Page 5, in paragraph 7 line 5, the following sentences should be added to the term of detection limit. "The minimum acceptance number of blank samples should be included in definition (eg:>20).

·Page 5, in paragraph 9 line 1, LOQ definition should use either 6s or 10s (both criteria can not be used).

UNITED STATES OF AMERICA

The United States fully supports the author's use of definitions from ISO 5725-1 for many of the terms used in this paper. We also submit the following specific comments on various sections of Annex 1 of CL 2003/43-MAS for consideration.

In the **Result** section, we recommend that the bullet containing the corrected result be modified as follows: "the corrected result (including the manner of correction, e.g., using an internal standard added at the beginning of the analysis); and". This modification will provide additional valuable information to the users of the results, especially for LC/MS and GC/MS methods that commonly recommend internal standards.

In the **Specificity** section, we believe that the statement, "In some cases specificity is not desired (e.g., total fat, fatty acids, crude protein, dietary fiber, reducing sugars)." could be misleading. Even though some methods are not necessarily specific, it is important that the methods not be influenced or affected by non-target analytes. We recommend this note be modified as follows: "In some cases specificity is not desired (e.g., total fat, fatty acids, crude protein, dietary fiber, reducing sugars), but the method must not be affected by interferences or non-target analytes."

In the **Accuracy (as a concept) and Accuracy (as a statistic)** section**,** we suggest that making a distinction between accuracy as a concept and accuracy as a statistic goes beyond the ISO 5725-1 definition and may create unnecessary confusion. The authors make the distinction that "Accuracy as a statistic applies to a single reported final test result; accuracy as a concept applies to single, replicate or averaged values." We consider this distinction to be unnecessary and recommend that the ISO 5725-1 definition for accuracy be used.

In the **Reproducibility Conditions** section, we think that the note under this definition allowing multiple methods to be used with respect to "Reproducibility conditions" is a significant departure from accepted definitions for this term. The definition of "Reproducibility conditions" as it applies to the same method on identical test items with different laboratories with different operating conditions using different equipment has generally been accepted by ISO, IUPAC and AOAC. Expanding the definition to accommodate proficiency studies or material certification studies should be examined very carefully. We suggest that a term be developed specifically to address proficiency studies or material certification studies separate from Reproducibility conditions.

In the **Detection Limit and Determination Limit** sections, we believe that the definitions provided in this paper for Detection Limit and Determination Limit are somewhat convoluted. We suggest that the Committee consider the latest efforts in harmonizing analytical terminology from international organizations before deciding on definitions.

In the **Linearity** section, the word quality in the first sentence should be **quantity**. The sentence should read: "The ability of a method of analysis, within a certain range, to provide an instrumental response or results proportional to the **quantity** of analyte to be determined in the laboratory sample."