codex alimentarius commission

FOOD AND AGRICULTURE ORGANIZATION OF THE UNITED NATIONS

WORLD HEALTH ORGANIZATION

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Agenda Item 2a) **CX/FFP 03/2-Add.2**

JOINT FAO/WHO FOOD STANDARDS PROGRAMME
CODEX COMMITTEE ON FISH AND FISHERY PRODUCTS

Twenty-sixth Session
Ålesund, Norway, 13 - 17 October 2003

MATTERS REFERRED TO THE COMMITTEE BY THE CODEX ALIMENTARIUS COMMISSION AND OTHER CODEX COMMITTEES

A. COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

Endorsement of methods of analysis

The 24th Session of the Committee on Methods of Analysis and Sampling did not endorse the following methods in the Standard for Boiled Dried Salted Anchovies (ALINORM 03/23, Appendix VI):

- water activity (method AOAC 978.18) : CCFFP was asked to provide clarification as the method proposed applies to canned vegetables
- Acid Insoluble Ash (method described in the standard) : CCFFP should provide information on the validation of the method

The Committee is invited to provide additional information on these methods in order to facilitate further consideration and endorsement in the next session of the Committee on Methods of Analysis and Sampling.

General issues

The 24th Session of the Committee on Methods of Analysis and Sampling considered *The Use of Analytical Results: Sampling, Relationship Between the Analytical Results, the Measurement Uncertainty, Recovery Factors and the Provisions in Codex Standards* on the basis of a document prepared by the United Kingdom.

The Delegation of the United Kingdom indicated that decisions regarding the acceptability of a lot or sample should be based on a concept that takes sampling and analytical aspects into consideration. The Delegation pointed out that at the present time there was no common understanding and interpretation of analytical results among Codex Members and therefore different decisions might be taken after an analysis of the same sample. This occurred because some countries took into account uncertainty for the interpretation of results while others did not and different sampling regimes were used. The Delegation proposed that when Commodity Committees develop specifications they should do it with respect to those factors which affect the interpretation of specifications. Therefore Commodity Committees should give clear guidance to the Committee on Methods of Analysis and Sampling on how they wished Codex specifications to be enforced.

Many delegations emphasized the importance of this issue in order to ensure consistency throughout Codex and supported efforts in this area. It was also suggested to include recommendations to Commodity Committees in the Procedural Manual in order to ensure a consistent approach throughout Codex. Some delegations were of the view that before proceeding further this problem should be addressed by Commodity Committees as they should consider how the analytical results would be used when developing provisions in Codex Standards.

The Committee agreed to forward the working document to Commodity Committees and to the Committee on Food Import and Export Inspection and Certification Systems for consideration and comments (ALINORM 03/23, paras. 109-117). The document is presented in **Annex 1** for consideration by the Committee .

B. COMMITTEE ON ADDITIVES AND CONTAMINANTS

Draft Maximum Level for Lead in Fish

The 34th Session of the CCFAC (2002) decided that the draft maximum level of 0.2 mg/kg for lead in fish, as well as the list of certain species for which the level might not apply, should be returned to Step 6 for comments and further consideration. The 35th Session (2003) noted the suggestion of a two-tiered approach, namely, the establishment of a limited list of internationally traded fish species that could comply with a level of 0.2 mg/kg and, the establishment of a limited list of internationally traded fish species that could comply with a level 0.4 mg/kg. In any case, the Committee noted that it should focus its efforts on those species that were significantly traded internationally and that specific scientific species names were required.

Several delegations expressed concern about this approach, as short positive lists with the corresponding levels could actually create barriers to trade for those species excluded from the lists. These delegations expressed preference for one level that was practically achievable and based on the data submitted, i.e., 0.5 mg/kg. These delegations also explained that the available analytical equipment in their countries could measure a level of 0.5 mg/kg, as opposed to technical and economic difficulties in measuring lower levels.

The Committee could not reach a consensus on this issue and therefore, decided to return the draft maximum level (Appendix XIII of ALINORM 03/12) to Step 6 for comments and further consideration at its 36th Session. The Committee agreed that in the interim, a statistical analysis should be performed based on the comments submitted and additional data available (GEMS Food, FAO) using different levels of concern (e.g., 0.2, 0.4 and 0.5 mg/kg) as a basis for making a decision on whether or not to adopt a tiered approach. It was noted that the analysis should provide information on the percentage of rejected samples using different maximum levels for species traded internationally in significant quantities.

The delegation of Denmark stressed the need for more data and information about fish species traded internationally. In this regard, it was noted that data should be forwarded in GEMS Food format. The Committee accepted the offer of the delegation of Denmark to collect the data and to do a statistical analysis of data on lead content for significantly traded fish species (identified by Latin names) that might cause problems in international trade (e.g. tuna, salmon, mackerel, cod, herring, pollack and sardines) (ALINORM 03/13A, paras. 137-142).

C. MATTERS FROM FAO AND WHO

Joint FAO/WHO Expert Committee on Food Additives - Methylmercury

The 61st Meeting of the Joint FAO/WHO Expert Committee on Food Additives (JECFA) (Rome, 10-19 June 2003) re-evaluated methylmercury, and established a Provisional Tolerable Weekly Intake (PTWI) of 1.6 µg/kg bodyweight. The Summary and Conclusions of the 61st JECFA are available on the FAO and WHO websites at www.fao.org/es/esn/jecfa/index_en.stm and www.who.int/pcs/jecfa/jecfa.htm

Background information

The background and current situation as regards methylmercury in fish in the framework of Codex is the following.

The current Guideline Levels for methylmercury in fish adopted by the 19th Session of the Commission (1991) are: 0.5 mg.kg in all fish except predatory fish and 1 mg/kg in predatory fish. At the request of the Commission, the Committee on Fish and Fishery Products initiated work on the development of a list of predatory fish. This question was considered by the 20th, 21st and 22nd sessions (1992 to 1996). The 22nd Session agreed to inform the Executive Committee, the Commission and Committee on Food Additives and Contaminants of its discussions and of the difficulties identified in the development of a list.

The 43rd Session of the Executive Committee (1996) recommended that a new risk analysis be undertaken, including an evaluation of newly available information, with consideration being given to the establishment of new risk management options as part of the Codex Guideline, particularly any action relevant to the current Guideline. The CCEXEC asked the Committee on Food Additives and Contaminants to initiate the necessary work. The 29th Session of the CCFAC (1997) agreed to defer any decision on this matter until JECFA had performed the necessary risk assessment (ALINORM 97/12A, para. 6) and it was not discussed further in the following sessions.

Agenda Item 9 **CX/MAS 02/13**

JOINT FAO/WHO FOOD STANDARDS PROGRAMME
CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

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

THE USE OF ANALYTICAL RESULTS: SAMPLING, RELATIONSHIP BETWEEN THE ANALYTICAL RESULTS, THE MEASUREMENT UNCERTAINTY, RECOVERY FACTORS AND THE PROVISIONS IN CODEX STANDARDS

(Prepared by the United Kingdom)

INTRODUCTION

It was noted at the 23rd Session of the Codex Committee on Methods of Analysis and Sampling (CCMAS) that there were a number of decisions that may be taken by those responsible for the enforcement of Codex specifications which directly affect decisions as to whether a lot is in compliance with a Codex specification (see ALINORM 01/23, paras 60 and 64).

It was therefore proposed that a paper be prepared outlining the issues involved. This paper describes the issues and makes recommendations and guidance to governments that could be included in Volume 13 of the Codex to aid the development and subsequent enforcement of Codex Commodity standards.

This paper is written in a form such that the issues identified could be readily appreciated by Codex Commodity Committees.

Issues Involved

There are a number of analytical and sampling considerations which prevent the uniform implementation of legislative standards; these are addressed in this paper. In particular the problems of:

1. the basic principles of the sampling procedures used by the Member States of Codex to enforce Codex Standards (see Annex I)
2. the treatment of analytical variability (normally known as the measurement uncertainty) in the interpretation of a Codex specification (see Annex II), and
3. the use of recovery corrections when calculating and reporting analytical results (see Annex III).

are addressed in the Annexes. The effect of different countries taking different approaches for each of the issues identified are described.

It must be appreciated that there may be other enforcement issues which have a similar effect.

These aspects directly affect the interpretation of results in countries which use Codex Standards and so may be regarded as "food control". At the present time there is no common interpretation of analytical results across the Codex Community so significantly different decisions may be taken after analysis of the "same sample". Material for which there is a statutory limit of, say, 4μg/kg for a contaminant may be interpreted as containing 3μg/kg on analysis in one country but 10μg/kg in another. This is because some countries correct analytical results for recovery, others do not; some countries use an "every-item-must-comply" sampling regime, others may use an "average of a lot" regime.

It is essential that interpretation of analytical results is similar if there is to be equivalence across the Codex Community; without it there is no uniform interpretation of Codex standards.

It is stressed that this is not an analysis or sampling problem as such but an administrative problem which has been highlighted as the result of recent activities in the analytical sector, most notably the development of International Guidelines on the Use of Recovery Factors when Reporting Analytical Results, and various Guides prepared dealing with Measurement Uncertainty.

The effects are addressed in the Annexes to this paper.

SOLUTION

It is important that delegates to Codex Commodity Committees realise that different actions taken with respect to the above consideration have a significant difference on the "enforcement" of the Codex Provisions. Because the effect is so marked, it is important that delegates to Commodity Committees are aware that there is the possibility that different countries will "interpret" the commodity standard with respect to compliance of a lot in different ways. It is therefore recommended that when Codex Commodity Committees negotiate specifications they do so with respect to those factors which affect the interpretation of the Codex specification. In addition the Commodity Committee should give clear simple guidance to CCMAS with respect to how it wishes the Codex specification to be "enforced". This guidance is to cover both sampling plans and aspects of the analytical enforcement of the commodity specifications.

RECOMMENDATIONS

It is recommended that at the same time that the Codex Commodity Committee discusses and agrees a commodity specification, it states the following information:

Sampling

The principle on which any sampling plans are to be developed, and in particular whether any detailed plans subsequently developed by CCMAS are to be on the basis that the specification applies to every item in a lot or to the average in a lot, and the appropriate acceptable quality level to be used.

Measurement Uncertainty

Whether allowance for the measurement uncertainty is to be made when deciding whether an analytical result falls within the specification or not.

Recovery

Whether the analytical result of a lot is to be reported on a recovery corrected or uncorrected basis.

Although each of the above attracts a number of scientific considerations, it is of prime importance that all Codex countries adopt the same approach so that a common approach to enforcement of Codex standards is taken.

ANNEX I: INFORMATION FOR CODEX COMMODITY COMMITTEES ON THE SELECTION OF CODEX SAMPLING PROCEDURES AND INTERPRETATION OF CODEX SPECIFICATIONS

INTRODUCTION AND GENERAL BACKGROUND

Codex sampling plans are designed to ensure that fair and valid procedures are used when food is being tested for compliance with a particular Codex commodity standard. The sampling procedures are intended for use as international methods designed to avoid or remove difficulties which may be created by diverging legal, administrative and technical approaches to sampling and by diverging interpretation of results of analysis in the light of the relevant provision(s) of the applicable Codex Standard.

Codex Committees should, when developing provisions (characteristics) in a Standard, relate the numerical value of the characteristic, the associated method of sampling and the method of analysis to one another. The Codex General Principles for Analysis and Sampling (Codex Alimentarius Commission, Procedural Manual, Tenth Edition) are intended to ensure that this will be done when selecting Codex methods of sampling and analysis for inclusion in Codex Standards. This requirement is generally followed when methods of analysis are to be developed but, regretfully, infrequently when methods of sampling are to be elaborated.

This is generally because the importance of the relationship is not always understood or is considered to be too complex; this paper is intended to demonstrate that the significance of the relationship and thus encourage Codex Commodity Committees to address the sampling requirements in their Standards.

Specification Limit and Interpretation of Results

It is important that a Codex Commodity Committee considers and then defines exactly how the specification is to be interpreted. Without this information it is difficult to develop the methods of sampling and analysis which are then to be used to interpret the specification. This may be best illustrated by the example below:

Let us assume that a lot of 1,000 units of, say, a foodstuff is to be investigated to ascertain whether it is in compliance with a Codex specification of 2 mg/kg lead.

If each of the 1,000 units were to be sampled and analysed for its lead content, then the distribution of lead in the individual units may be shown diagrammatically below:

Figure: plot of the distribution of lead in the 1,000 units, with minimum concentration of 1.5 mg/kg, mean concentration in the lot of 1.9 mg/kg and maximum concentration of 2.3 mg/kg. The specification limit is 2 mg/kg.

Two countries may have different national rules for the interpretation of results from lots.

Country A requires: that each and every item in the lot meets the specification. In this example it means that all 1,000 units, if analysed separately, would have to be less than 2.0 mg/kg. Here a significant number of units are greater than 2.0 mg/kg so the lot would be deemed to be in non-compliance with the Codex specification and so would be rejected, but

Country B requires: that the mean value of the characteristic in the lot is to be less than the Codex specification. In this case the mean value is 1.9 mg/kg so the lot would be deemed to be in compliance with the Codex specification.

Consequence:	the two countries A and B will make different judgements as to compliance with a Codex specification on essentially the same lot. This is unacceptable and can only be avoided if the sampling procedures are elaborated at the same time as the commodity standard is elaborated in the Commodity Committee. In addition it should also be noted that the number of units to be analysed also influences the decision on compliance (see below).

The approach to be taken must be defined before any sampling procedure is discussed. At present there is no information given as to the basis on which the Codex specification is to be evaluated prior to discussions on sampling commencing. This creates severe difficulties when methods of sampling are developed. The procedure for the analysis of the individual sample units is now well defined within Codex, but the framework within which the results are to be used is not.

Relationship Between Value of a Characteristic in a Commodity Standard and Methods of Analysis and Sampling Used for its Estimation

Before any characteristic in any Codex Standard is elaborated it must be appreciated that the value of the characteristic in that Codex Standard is dependent on the procedures used to estimate that value. In particular, the estimate of the value may be dependent upon the method of analysis used, but is always dependent on the method of sampling used to verify compliance with the Standard. It is important for delegates at Codex Commodity Committees to appreciate the influence that methods of analysis and sampling may have on the judgements that may be made with regard to the compliance of a lot with respect to a Codex Commodity Standard. Without common and uniform methods of analysis and sampling procedures different authorities will make different judgements as to whether any particular lot is compliance with its Codex specification, as has been illustrated above. The relationship between the value of a characteristic in a Codex Commodity Standard and the method of analysis to estimate that value can be readily appreciated, but the link between the value of the characteristic and the method of sampling is less well understood.

This is best illustrated by example, taking first methods of analysis, and then methods of sampling.

Methods of Analysis

This may be best illustrated by reference to the "types" of methods of analysis which have been adopted by the Codex Alimentarius Commission. The CAC has stated that as Type I methods "define" the value of the characteristics in the Standard only a single Type I method can be prescribed. Methods of analysis for "fat" are Type I methods. It is possible to determine the "fat" content in a sample by two equally validated methods of analysis, each conforming to a different analytical principle. As a consequence the application of these two methods to the same sample will result in two different, but equally valid, results. In order to remove this possibility the Codex system only allows the adoption of a single Type I method.

In addition it is a mandatory requirement to accept the Type I Codex method if the Standard itself is to be accepted - i.e. the separation of the value of the characteristic and the relevant Type I method is, in effect, meaningless. It has, therefore, been agreed by the Codex Committees on Methods of Analysis and Sampling and on General Principles that non-acceptance of the Codex defining methods, or acceptance of Codex Standards with substantial deviations in the Codex defining method, should be taken to mean acceptance of the Codex Standard with a specified deviation.

Codex Type II and III methods determine the content of a defined chemical entity and these methods may be used interchangeably depending upon the particular situation except that Type II Codex methods are intended to be obligatory in cases of disputes concerning the results of analysis. However this approach may be modified as a result of the present discussions on the introduction of a criteria (performance-based) approach to methods of analysis in Codex Commodity Standards.

Methods of Sampling

The same considerations as apply to methods of analysis also apply to methods of sampling. This may also be best illustrated by a simple example.

One of the criteria by which the quality of a lot may be judged is the acceptable quality level (AQL) for a specification in a lot. In simple terms, the acceptable quality level in a lot is the percentage of defective items that is considered satisfactory as a process average and is accepted with a given high probability of acceptance (usually in the region of 95%). For a specification in a batch two countries may have different acceptable quality levels i.e.

Country A may prescribe an acceptable quality level of 0.1%, i.e. it will only accept a batch if 99.9% of the product meets the specification whereas

Country B has prescribed an AQL of 10%, i.e. that country will accept the batch if 90% of the product meets the specification.

The amount of sampling and the commodity specification required to determine these two batches is different in each case and thus there is no harmonisation of sampling. If left undefined these two countries could make different judgements as to whether a particular lot would comply with a Codex specification.

One of the critical aspects of sampling is that numbers of units must be taken at random throughout the batch. This is often difficult to achieve and the approach to randomisation will produce different decisions as to compliance or non-compliance of a batch. It is therefore important that if a uniform approach to sampling is to be taken, that procedures for randomisation are carefully defined.

This, and similar, procedures must be defined **before** sampling plans are discussed.

TIMING OF THE DEVELOPMENT OF SAMPLING AND ANALYSIS PROCEDURES

It has been illustrated above that the type of sampling plan and the lot acceptance procedure used affects whether a lot may be deemed to be in compliance with its specification. It is therefore necessary that when characteristics within a Standard are elaborated, the sampling and lot acceptance procedures to be prescribed to verify those characteristics are also considered at the same time, so that the characteristics are related to the procedures.

It is important to recognise that without general instructions being given to those preparing Codex sampling plans, non-equivalent interpretation of Codex Commodity Standards will occur, thus giving the potential for trade disputes.

To define a numeric value in a Standard is not enough: its interpretation also needs to be defined.

ANNEX II: REPORTING OF RESULTS WITH RESPECT TO THEIR MEASUREMENT UNCERTAINTY

All analytical results should be reported in the form "a ± b" where "a" is the best estimate of the true value of the concentration of the measurand (the analytical result) and "b" is the range within which the true value is estimated, with a given probability, to fall. The value of "b" is known as the "measurement uncertainty" and may be estimated by the analyst in a number of different ways. Even though this terminology is considered suspect by some, it is now internationally accepted.

The estimation of the value of "a" is dependent on:

- the accuracy of the method of analysis used
- how well the analyst uses that method, i.e. whether the analytical system is "in control".

The value of the measurement uncertainty "b" is dependent on:

- the inherent precision of the method of analysis used
- the number of analytical replicates that are carried out. The more replicates the less the value of the measurement uncertainty.

REPORTING OF RESULTS BY FOOD CONTROL ANALYSTS

The procedure adopted by some food control analysts is to report samples as containing "not less than "a" – "b"" in situations where the statutory limit is a maximum permissible concentration. Thus, in any enforcement situation the maximum benefit is given to the food producer. This is consistent with the requirement to prove ***beyond reasonable doubt*** that a limit has been exceeded, if the case should come to Court. This does mean that the effective enforcement limit is, in such countries, not identical to the numerical value given in the Codex specification.

Other food analysts may report the value "a" without taking into account any measurement uncertainty considerations.

CONSEQUENCES OF REPORTING RESULTS IN DIFFERENT WAYS

There are potential problems with the reporting of results for which there is a Codex specification.

This is best explained by example:

Let us assume that there is a Codex specification of 4 μg/kg for the analyte being analysed. It would be anticipated that the measurement uncertainty for the analysis will be of the order ± 45% of the analytical result, i.e. the analyst would determine for nominal concentrations of 3, 6 and 10 μg/kg, the following concentrations including their uncertainties:

a. 3.0 ± 1.3 μg/kg,

b. 6.0 ± 2.6 μg/kg, and

c. 10.0 ± 4.4 μg/kg

Situation a

Here the level reported is below the Codex specification. All countries would take the same view and accept the material.

Situation b

Here the level reported is above the statutory limit but the true value lies in the range 3.4 to 8.6 μg/kg. The level and its uncertainty would be reported.

Here some countries would report the sample as containing not less than 3.4 μg/kg of the analyte and because it is not beyond reasonable doubt that the limit has been exceeded, no action will be taken.

However, other countries may take action on the 6.0 μg/kg result, without taking uncertainty into account. For these countries, the material will be deemed to be non-compliant.

Situation c

Here the level reported is above the Codex specification and the true value lies in the range 5.6 to 14.4 μg/kg. All countries will state that the material is non-compliant with the Codex specification.

Conclusion

In situation b there is the possibility that different countries will make opposite decisions as to whether the material conforms with the Codex specification. The approach to be used must be indicated by the Codex Commodity Committee when negotiating the Codex Commodity Standard.

ANNEX III: USE OF RECOVERY INFORMATION IN ANALYTICAL MEASUREMENT

CCMAS has discussed the harmonisation of reporting of test results corrected for recovery factors. In particular it has adopted by reference the "Harmonised Guidelines for the Use of Recovery Information in Analytical Measurement", published by IUPAC. However, it did not adopt by reference the first two sentences of the first Recommendation, namely "Quantitative analytical results should be corrected for recovery unless there are specific reasons for not doing so. Reasons for not estimating or using correction factors include the situations where (a) the analytical method is regarded as empirical, (b) a contractual or statutory limit has been established using uncorrected data, or (c) recoveries are known to be close to unity."

The next three sentences of Recommendation 1 are also important, these being:

"However, it is of over-riding importance that all data, when reported, should (a) be clearly identified as to whether or not a recovery correction has been applied and (b) if a recovery correction has been applied, the amount of the correction and the method by which it was derived should be included with the report. This will promote direct comparability of data sets. Correction functions should be established on the basis of appropriate statistical considerations, documented, archived and available to the client."

The above serves to indicate the importance of recovery corrections and, as in the previous Annexes, one can obtain a similar situation where different countries may report a different analytical result depending upon whether a recovery correction has been made or not.

A real example may result in the mycotoxin area where there may be a limit of 4μg/kg for total aflatoxin in nuts. Here the following situation may arise: Country A will analyse a consignment and find a result of 3.5μg/kg total aflatoxin using a method which, in the analytical run, has a recovery of 70%. Country A does not correct for recovery corrections as a matter of policy and so the reported result will be 3.5μg/kg and so the sample will be in compliance with the 4μg/kg limit.

Country B, however, uses recovery corrections as a matter of policy. That country could analyse the "same" sample using the "same" methodology and obtain the "same" analytical result but will report not 3.5 but 5μg/kg on a recovered basis. Here there is the possibility that because the 5μg/kg level is greater than the Codex limit of 4μg/kg limit for total aflatoxin that country may deem the sample not to be in compliance with the Codex limit.

As in the previous situations it is important that the Codex Commodity Committee stipulates the basis on which the Codex specification is to be enforced.