codex alimentarius commission

FOOD AND AGRICULTURE ORGANIZATION OF THE UNITED NATIONS

WORLD HEALTH ORGANIZATION

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Agenda Item 2 **CX/MAS 01/2**

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING
Twenty-Third Session
Budapest, Hungary, 26 February - 2 March 2001

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

A. MATTERS ARISING FROM THE CODEX ALIMENTARIUS COMMISSION

The Commission did not consider draft texts or questions arising from the CCMAS at its 23rd Session. The following question was referred both to the Committee on Additives and Contaminants and to the CCMAS.

Draft Maximum Level and Sampling Plans for Total Aflatoxins in Peanuts Intended for Further Processing

The Commission adopted the maximum level of 15µg/kg and sampling plans in peanuts for further processing. While discussing this issue, some delegations proposed an alternative sampling plan in order to address more thoroughly sample selection, sample preparation and analytical methods for the detection of aflatoxins. It was noted that the maximum level and sampling plans were developed on the basis of an FAO Expert Consultation[1] and an extensive risk assessment was recently conducted by the 49th Session of JECFA.
The Commission adopted the draft sampling plan on an interim basis, with the understanding that the issue would be further considered by the Committee (CCFAC) and the Committee on Methods of Analysis and Sampling on the basis of proposals to be developed by an electronic working group prior to their next sessions (ALINORM 99/37, paras. 100-102).

B. MATTERS ARISING FROM OTHER CODEX COMMITTEES

1. Codex Committee on Food Additives and Contaminants

Sampling Plan for Aflatoxins

The 32nd Session of the Committee (2000) considered the need to revise the Sampling Plans for Aflatoxins, in view of the decision of the Commission. The Committee noted that the 23rd Session of CCMAS was not scheduled to be held until late February 2001, immediately prior to the 33rd CCFAC. The Committee therefore decided that a drafting group would prepare a proposed draft revision of the sampling plan for peanuts for circulation, comment and consideration at its next meeting. It was further decided that the sampling plan should also be referred for consideration by the 23rd Session of the CCMAS (ALINORM 01/12, para. 10).

The text of the Proposed Draft Revised Sampling Plan for Peanuts was circulated for comments at Step 3 in document CX/FAC 01/21 and is attached in **Annex I** for consideration by CCMAS.

Methods of analysis for additives and contaminants

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1 FAO Consultation on Sampling Plans for Aflatoxin Analysis in Peanuts and Corn (FAO Food and Nutrition paper 55, 1993)

The 31st Session of the CCFAC agreed to forward proposals concerning the determination of lead, cadmium, zinc, copper and iron to the CCMAS for consideration (NKML method). This method was included in the document on Endorsement of Methods (CX/MAS 01/10) and will be considered together with the other methods proposed by Codex Committees under **Agenda Item 9**.

Dioxins

The 32nd Session of CCFAC considered a paper on dioxins and agreed to inform the Task Force on Animal Feeding and the CCMAS of its discussion as a matter of interest. The relevant section of ALINORM 01/12 is included in **Annex II**. The Committee requested the CCMAS to provide information on methods of analysis for dioxins.

2. Codex Committee on General Principles

The matters relevant to the discussion on Criteria will be considered under **Agenda Item 4** and the recommendations of the CCGP in this area will be included in a separate document (CX/MAS 01/5-Add.1).

3. Codex Committee on Food Labelling

The Committee on Food Labelling is currently discussing the labelling of foods obtained through certain techniques of genetic modification/ genetic engineering, as an amendment to the General Standard for the Labelling of Prepackaged Foods. While discussing the inclusion of threshold levels, several delegations pointed out that analytical methods should be considered by the CCMAS. It was noted that the *Ad hoc* Intergovernmental Task Force on Foods Derived from Biotechnology had decided to discuss this issue at its next session in March 2001. The Committee recognized the importance of close collaboration among Codex bodies and decided to ask the CCMAS to study the methods for the detection or identification of food and food ingredients derived from biotechnology (ALINORM 01/22, para.44).

The notes of the CCMAS Agenda indicate that the Committee will consider this request, taking into account the preliminary list of methods prepared in the framework of the *Ad hoc* Intergovernmental Task Force on Foods Derived from Biotechnology. Information on methods was requested in the framework of the Task Force by Circular Letter CL 2000/29-FBT/MAS (September 2000). It was expected that the document prepared for the Task Force would be available for consideration by CCMAS; however, it has not yet been finalized.

The Committee is invited to consider how it wishes to proceed in this area in view of the request from the Committee on Food Labelling.

4. Codex Committee on Fish and Fishery Products

Some matters were referred back by the CCFFP, following earlier questions from the CCMAS on specific methods of analysis. They are included in the document on Endorsement (CX/MAS 01/10) as they relate to methods submitted for endorsement, for consideration under **Agenda Item 9**.

5. Codex Committee on Processed Fruits and Vegetables

Net Drained Weight

The last session of the CCMAS noted that the 19th Session of the CCPFFV requested advice on the tolerances permitted for the declaration of drained weight (following discussion in the Standard for Canned Pears). Noting that this was rather a technological problem and that it would not seem feasible to establish general tolerances for net drained weight, the Committee agreed to refer it back to CCPFFV (ALINORM 99/23, para. 6).
The 20th Session of the CCPFFV advanced the Draft Revised Standard for Canned Pears to Step 8 and the Weight and Measures Section was left unchanged.

Methods of Analysis

The CCPFFV did not discuss the specific methods of analysis in the standards under elaboration. It agreed to forward to the CCMAS the working document presented to the Committee (CX/PFV 00/7) for endorsement along with the information presented in the written comments (ALINORM 01/27, para.42). However, this does not allow to establish sections on methods for the standards under consideration by the Committee. The document includes methods for standards which are not yet under consideration by the Committee as well as for standards under elaboration in the Step Procedure.

The following Draft Standards were forwarded to Step 8: Canned Applesauce, Canned Pears, Kimchi.

- The additional method mentioned in the CCPFFV document concerning Canned Applesauce was included in the Endorsement paper (CX/MAS 01/10).

- The methods in the Draft Standard for Kimchi were already endorsed previously

- No specific method was proposed for Canned Pears (general methods are applicable).

In the Draft Standard for Pickles, a number of questions were raised by the last session of the CCMAS, as indicated in the Endorsement paper (CX/MAS 01/10). As the CCPFFV returned the Draft Standard for Pickles to Step 6, that will allow for further consideration of the methods at the next session.

For the standards returned to Step 6 or forwarded to Step 5, the CCPFFV may be invited to establish sections on methods for each standard, in conformity with the format of Codex Standards, and also to indicate the amendments to be made to general methods, if required.

ANNEX I

PROPOSED DRAFT SAMPLING PLAN FOR TOTAL AFLATOXINS IN PEANUTS INTENDED FOR FURTHER PROCESSING TO BE USED FOR ENFORCEMENT AND CONTROL PURPOSES

INTRODUCTION

The 23rd Session of the Codex Alimentarius Commission adopted the sampling plan for "Total Aflatoxins in Peanuts Intended for Further Processing" on an interim basis. The sampling plan calls for a single 20 kg laboratory sample to be taken from a peanut lot and tested against a maximum level of 15 parts per billion total aflatoxin.

This sampling plan has been designed for enforcement and controls concerning total aflatoxins in bulk consignments of peanuts traded in the export market. To assist member countries in implementing the Codex sampling plan, sample selection methods, sample preparation methods , and analytical methods required to quantify aflatoxin in bulk peanut lots are described in this document.

A. Definitions

Lot : an identifiable quantity of a food commodity delivered at one time and determined by the official to have common characteristics, such as origin, variety, type of packing, packer, consignor or markings.

Sublot: designated part of a large lot in order to apply the sampling method on that designated part. Each sublot must be physically separate and identifiable.

Sampling plan: is defined by an aflatoxin test procedure and an accept/reject limit. An aflatoxin test procedure consists of three steps: sample selection, sample preparation, and aflatoxin quantification. The accept/reject limit is a tolerance usually equal to the Codex maximum limit.

Incremental sample: a quantity of material taken from a single random place in the lot or sublot.

Aggregate sample: the combined total of all the incremental samples taken from the lot or sublot. The aggregate sample has to be at least as large as the 20 kg laboratory sample.

Laboratory sample: smallest quantity of peanuts comminuted in a mill. The laboratory sample may be a portion of or the entire aggregate sample. If the aggregate sample is larger than 20 kg, a 20 kg laboratory sample should be removed in a random manner from the aggregate sample.

Test portion: portion of the comminuted laboratory sample. The entire 20 kg laboratory sample should be comminuted in a mill. A portion of the comminuted 20 kg sample is randomly removed for the extraction of the aflatoxin for chemical analysis. Based upon grinder capacity, the 20 kg aggregate sample can be divided into several equal sized samples, if all results are averaged.

B. Sampling

Material to be sampled

Each lot, which is to be examined, must be sampled separately. Large lots should be subdivided into sublots, to be sampled separately. The subdivision can be done following provisions laid down in the table hereafter.

Taking into account that the weight of the lot is not always an exact multiple of the weight of the sublots, the weight of the sublot may exceed the mentioned weight by a maximum of 20 %.

Table : Subdivision of large lots into sublots for the sampling

Commodity	Lot weight - tonne (T)	Weight or number of sublots	N° incremental samples	Laboratory sample Weight (kg)
Groundnuts	≥ 500	100 tonnes	100	20
	>100 and <500	5 sublots	100	20
	≥ 25 and ≤ 100	25 tonnes	100	20
	>15 and <= 25	--1 sublot	100	20

Number of incremental samples for lots of less than 15 tonnes

The number of incremental samples to be taken depends on the weight of the lot, with a minimum of 10 and a maximum of 100. The figures in the following table may be used to determine the number of incremental samples to be taken.

Table: Number of incremental samples to be taken depending on the weight of the lot.

Lot weight tonnes – (T)	**N° of incremental samples**
T≤ 1	10
1 <T ≤ 5	40
5< T ≤ 10	60
10<T < 15	80

Incremental Sample Selection

Procedures used to take incremental samples from a peanut lot are extremely important. Every individual peanut in the lot should have an equal chance of being chosen. Biases will be introduced by the sample selection methods if equipment and procedures used to select the incremental samples prohibit or reduce the chances of any item in the lot from being chosen.
Since there is no way to know if the contaminated peanut kernels are uniformly dispersed through out the lot, it is essential that the aggregate sample be the accumulation of many small portions or increments of the product selected from different locations throughout the lot. If the aggregate sample is larger than desired, it should be blended and subdivided until the desired laboratory sample size is achieved.

Static Lots

A static lot can be defined as a large mass of peanuts contained either in a single large container such as a wagon, truck, or railcard or in many small containers such as sacks or boxes and the peanuts are stationary at the time a sample is selected. Selecting a truly random sample from a static lot can be difficult because the container may not allow access to all peanuts.

Taking a aggregate sample from a static lot usually requires the use of probing devices to select product from the lot. The probing devices used should be specially designed for the type of container. The probe should (1) be long enough to reach all product, (2) not restrict any item in the lot from being selected, and (3) not alter the items in the lot. As mentioned above, the aggregate sample should be a composite from many small increments of product taken from many different locations throughout the lot.

For lots traded in individual packages, the sampling frequency, SF or number of packages that incremental samples are taken from is a function of the lot weight, LT, incremental sample weight, IS, aggregate sample weight, AS, and the individual packing weight, IP.

$$SF = (LT \times IS)/(AS \times IP) \qquad (1)$$

The sampling frequency, SF, is the number of packages sampled. All weights should be in the same mass units such as kg.

Dynamic Lots

True random sampling can be more nearly achieved when selecting an aggregate sample from a moving stream of peanuts as the lot is transferred, for example, by a conveyor belt from one location to another. When sampling from a moving stream, take small increments of product from the entire length of the moving stream; composite the peanuts to obtain an aggregate sample; if the aggregate sample is larger than the required laboratory sample, then blend and subdivide the aggregate sample to obtain the desired size laboratory sample.

Automatic sampling equipment such as cross-cut samplers are commercially available with timers that automatically pass a diverter cup through the moving stream at predetermined and uniform intervals. When automatic equipment is not available, a person can be assigned to manually pass a cup though the stream at periodic intervals to collect incremental samples. Whether using automatic or manual methods, small increments of peanuts should be collected and composited at frequent and uniform intervals throughout the entire time peanuts flow past the sampling point.

Cross-cut samplers should be installed in the following manner: (1) the plane of the opening of the diverter cup should be perpendicular to the direction of flow; (2) the diverter cup should pass through the entire cross sectional area of the stream; and (3) the opening of the diverter cup should be wide enough to accept all items of interest in the lot. As a general rule, the width of the diverter cup opening should be about three times the largest dimensions of the items in the lot.

The size of the aggregate sample, S in kg, taken from a lot by a cross cut sampler is

$$S = (D \times LT) / (T \times V) \qquad (2)$$

where D is the width of the diverter cup opening, cm, LT is the lot size in kg, T is interval or time between cup movement through the stream in seconds, and V is cup velocity in cm/sec. If the mass flow rate of the moving stream, MR (kg/sec), is known, then the sampling frequency, SF, or number of cuts made by the automatic sampler cup is

$$SF = (S \times V) / (D \times MR) \qquad (3)$$

Equation 2 can also be used to compute other terms of interest such as the time between cuts, T. For example, the required time, T, between cuts of the diverter cup to obtain a 20 kg aggregate sample from a 30,000 kg lot where the diverter cup width is 5.08 cm (2 inches), and the cup velocity through the stream 30 cm/sec. Solving for T in Equation 2,

$$T = (5.08 \text{ cm} \times 30{,}000 \text{ kg})/(20 \text{ kg} \times 30 \text{ cm/sec}) = 254 \text{ sec}$$

If the lot is moving at 500 kg per minute, the entire lot will pass through the sampler in 60 minutes and only 14 cuts (14 incremental samples) will be made by the cup through the lot. This may be considered too infrequent, in that too much product passes through the sampler between the time the cup cuts through the stream.

Weight of the incremental sample

The weight of the incremental sample should be about 200 grams.

Packaging and transmission of samples

Each laboratory sample shall be placed in a clean, inert container offering adequate protection from contamination and against damage in transit. All necessary precautions shall be taken to avoid any change in composition of the laboratory sample which might arise during transportation or storage.

Sealing and labelling of samples

Each laboratory sample taken for official use shall be sealed at the place of sampling and identified. A record must be kept of each sampling, permitting each lot to be identified unambiguously and giving the date and place of sampling together with any additional information likely to be of assistance to the analyst.

C. Sample preparation

Precautions

Daylight should be excluded as much as possible during the procedure, since aflatoxin gradually breaks down under the influence of ultra-violet light.

Homogenisation – grinding

As the distribution of aflatoxin is extremely non-homogeneous, samples should be prepared - and especially homogenised - with extreme care. All the material received by the laboratory is to be used for the homogenisation/grinding of the sample.
The sample should be finely ground and mixed thoroughly using a process that approaches as complete a homogenisation as possible.
The use of a hammer mill with a #14 screen (3.1 mm diameter hole in the screen) has been proven to represent a compromise in terms of cost and precision. A better homogenisation (finer grind – slurry) can be obtained by more sophisticated equipment, resulting in a lower sample preparation variance.

Test portion

A minimal test portion size of 100 g taken from the laboratory sample.

D. Analytical methods

Background

A criteria-based approach, whereby a set of performance criteria is established with which the analytical method used should comply, is appropriate. The criteria-based approach has the advantage that, by avoiding setting down specific details of the method used, developments in methodology can be exploited without having to reconsider or modify the specified method. The performance criteria established for methods should include all the parameters that need to be addressed by each laboratory such as the detection limit, repeatability coefficient of variation, reproducibility coefficient of variation, and the percent recovery necessary for various statutory limits. Utilising this approach, laboratories would be free to use the analytical method most appropriate for their facilities. Analytical methods that are accepted by chemists internationally (such as AOAC) may be used. These methods are regularly monitored and improved depending upon technology.

Performance criteria for methods of analysis

Specific requirements with which methods of analysis should comply

Criterion	Concentration Range	Recommended Value	Maximum permitted Value
Blanks	All	Negligible	-
Recovery-Aflatoxins Total	1 - 15 µg/kg	70 to 110 %	
	> 15 µg/kg	80 to 110 %	
Precision RSD_R	All	As derived from Horwitz Equation	2 x value derived from Horwitz Equation
Precision RSD_r may be calculated as 0.66 times Precision RSD_R at the concentration of interest			

- The detection limits of the methods used are not stated as the precision values are given at the concentrations of interest

- The precision values are calculated from the Horwitz equation, i.e.:

$$RSD_R = 2^{(1-0.5\log C)}$$

where:

* RSD_R is the relative standard deviation calculated from results generated under reproducibility conditions $[(s_R / \bar{x}) \times 100]$
* C is the concentration ratio (i.e. 1 = 100g/100g, 0.001 = 1,000 mg/kg)

This is a generalised precision equation which has been found to be independent of analyte and matrix but solely dependent on concentration for most routine methods of analysis.

ANNEX II

COMMITTEE ON ADDITIVES AND CONTAMINANTS, 32ND SESSION (2000): SECTION ON DIOXINS

(ALINORM 01/12, paras. 126-132)

DISCUSSION PAPER ON DIOXINS (Agenda Item 17d)

126. The 31st Session of the CCFAC requested the Netherlands to revise the Discussion Paper on Dioxins for circulation, comment and consideration at its current meeting.[2] The 23rd Session of the Codex Alimentarius Commission noted that work on dioxins had recommenced at the 31st Session of the CCFAC, and data was being sought to allow the establishment of an appropriate guideline or maximum level.[3] The Committee noted that dioxins and dioxin-like PCBs were on the CCFAC Priority List for JECFA evaluation.

127. Some delegations noted the absence of data on levels from many regions and rapid, cheap and reliable methods of analysis for dioxins and therefore, felt that it was premature to establish maximum levels. These delegations also noted that a reliable method of exposure assessment as well as the results of the JECFA evaluation were needed before proceeding further.

128. Other delegations, the representative of Consumers International and JECFA pointed out that WHO had undertaken a risk assessment in 1998 and that this could provide the basis for the elaboration of maximum levels and would provide industry and governments with a strong incentive to enforce source directed measures for the control of dioxins.

129. The JECFA Secretariat encouraged the submission of data on the types of foods and range of levels found in foods to allow the potential consideration of dioxins and dioxin-like PCBs at the 57th JECFA meeting in June 2001.

130. The Committee agreed that the delegation of the Netherlands would finalize the Discussion Paper and use it as a basis for the elaboration of a Position Paper on Dioxins and Dioxin-like PCBs. The Position Paper would include the potential range of levels in the commodities of interest (including feedingstuffs), explore the arguments for and against setting maximum limits and information on available methods of analyses, for consideration by the next Session of the CCFAC.

131. The Committee further agreed that Germany, in collaboration with Belgium, Japan, the Netherlands and the United States, would develop a proposed draft Code of Practice for Source Directed Measures to Reduce Dioxin Contamination of Foods for circulation, comment and consideration at its next meeting.

132. The Committee agreed to inform the *ad hoc* Intergovernmental Codex Task Force on Animal Feeding and the Codex Committee on Methods of Analysis and Sampling of the above discussions as a matter of interest. It also requested the Codex Committee on Methods of Analysis and Sampling to provide information on methods of analysis for dioxins.

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2 ALINORM 99/12A, para. 139

3 ALINORM 99/37, para. 236