CODEX ALIMENTARIUS COMMISSION E

Food and Agriculture Organization of the United Nations

World Health Organization

Viale delle Terme di Caracalla, 00153 Rome, Italy - Tel: (+39) 06 57051 - Fax: (+39) 06 5705 4593 - E-mail: codex@fao.org - www.codexalimentarius.org

Agenda Item 3 **CX/MAS 14/35/3**

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

Thirty-fifth Session
Budapest, Hungary, 3 - 7 March 2014

ENDORSEMENT OF METHODS OF ANALYSIS PROVISIONS IN CODEX STANDARDS

1. This document contains the methods of analysis and/or sampling proposed by the following Committees in draft standards and related texts under elaboration or as update of current methods:

PART I Methods of Analysis

A. Committee on Contaminants in Foods

PART II Methods of Sampling

A. Committee on Contaminants in Foods

PART I METHODS OF ANALYSIS

A. 7TH SESSION OF THE COMMITTEE ON CONTAMINANTS IN FOODS (CCCF)

2. See Table section A for the complete list of the proposed methods of analysis. Discussion at the Committee was as follows:

Proposed Draft Maximum Levels For Deoxynivalenol (DON) in Cereals and Cereal-Based Products and Associated Sampling Plans[1]

3. The CCCF agreed to include the performance criteria for methods of analysis, and to request advice from CCMAS on the appropriateness of the performance criteria to ensure consistency with the *Working Instructions for the Implementation of the Criteria Approach in Codex* (Procedural Manual).

PART II METHODS OF SAMPLING

A. 7TH SESSION OF THE COMMITTEE ON CONTAMINANTS IN FOODS (CCCF)

Proposed Draft Maximum Levels For Deoxynivalenol (DON) in Cereals and Cereal-Based Products and Associated Sampling Plans[2]

4. The CCCF agreed to forward the proposed draft MLs for raw cereal grains including sampling plans to Step 5. The 36th Commission adopted the proposal (See Annex I for the sampling plan).

1 REP12/CF para. 63
2 REP12/CF para. 70

A. COMMITTEE ON CONTAMINANTS IN FOODS

PROPOSED DRAFT MAXIMUM LEVELS FOR DEOXYNIVALENOL (DON) IN CEREALS AND CEREAL-BASED PRODUCTS AND ASSOCIATED SAMPLING PLANS - 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 specific 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 (within lab), reproducibility coefficient of variation (among lab), and the percent recovery necessary for various statutory limits. 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

A list of possible criteria and performance levels are shown in Table 3. Utilizing this approach, laboratories would be free to use the analytical method most appropriate for their facilities.

Table 3 Performance characteristics for deoxynivalenol

Level µg/kg	Deoxynivalenol		
	$RSD_{r\%}$	$RSD_{R\%}$	Recovery%
> 100 - ≤ 500	≤ 20	≤ 40	60 to 110
> 500	≤ 20	≤ 40	70 to 120

Note: Draft Maximum Levels for DON, adopted by the 36th CAC at Step 5, are as follows:

Product name	Maximum level (mg/kg)	Notes/Remarks
Raw cereal grains (wheat, maize and barley)	2	ML applies to raw cereal grains prior to sorting and removal of damaged kernels For sampling plan, see Annex below

ANNEX I

PROPOSED DRAFT SAMPLING PLANS FOR DEOXYNIVALENOL (DON) IN RAW CEREALS

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 larger 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 a deoxynivalenol test procedure and an accept/reject level. A deoxynivalenol test procedure consists of three steps: sample selection, sample preparation and analysis or deoxynivalenol quantification. The accept/reject level is a tolerance usually equal to the Codex maximum level (ML).

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

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

Laboratory sample – the smallest quantity of cereal/cereal based product comminuted in a mill. The laboratory sample may be a portion of or the entire aggregate sample. If the aggregate sample is larger than the laboratory sample(s), the laboratory sample(s) should be removed in a random manner from the aggregate sample.

Test portion – a portion of the comminuted laboratory sample. The entire laboratory sample should be comminuted in a mill. A portion of the comminuted laboratory sample is randomly removed for the extraction of the deoxynivalenol for chemical analysis.

Operating Characteristic (OC) Curve – a plot of the probability of a accepting a lot versus lot concentration for a specific sampling plan design. The OC curve provides an estimate of the chances of rejecting a good lot (exporter's risk) and the chances of accepting a bad lot accepted (importer's risk) by a specific deoxynivalenol sampling plan design. A good lot is defined as having a deoxynivalenol concentration below the ML; a bad lot is defined as having a deoxynivalenol concentration above the ML.

SAMPLE SELECTION

Material to be sampled

A) Sampling procedure for cereals and cereal products for lots ≥ 50 tonnes

Each lot, which is to be examined for deoxynivalenol must be sampled separately. Lots larger than 50 tonnes should be subdivided into sublots to be sampled separately. If a lot is greater than 50 tonnes, the lot has to be subdivided into sublots following Table 1

Table 1 Subdivision of lots into sublots depending on product and lot weight

Commodity	Lot weight (ton)	Weight or number of sublots	No incremental samples	Aggregate sample Weight (kg)
Raw wheat and barley	≥ 1 500	500 tonnes	100	1
	> 300 and < 1 500	3 sublots	100	1
	≥ 50 and ≤ 300	100 tonnes	100	1
	< 50	--	3-100*	1
Raw maize	≥ 1 500	500 tonnes	100	5
	> 300 and < 1 500	3 sublots	100	5
	≥ 50 and □ 300	100 tonnes	100	5
	< 50	--	3-100*	1-5

* Depending on the lot weight - see Table 2

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%.

- Each sublot must be sampled separately.

- Number of incremental samples: 100

- If it is not possible to carry out the method of sampling set out in this point because of the commercial consequences resulting from damage to the lot such as packaging forms, means of transport, an alternative method of sampling may be applied provided that it is as representative as possible and is fully described and documented.

Sampling procedure for cereals and cereal products for lots < 50 tonnes

For lots of cereals and cereal products less than 50 tonnes, the sampling plan must be used with 10 to 100 incremental samples, depending on the lot weight, resulting in an aggregate sample of 1 to 5 kg. For very small lots (≤ 0.5 tonnes) a lower number of incremental samples may be taken, but the aggregate sample uniting all incremental samples shall be also in that case at least 1 kg.

The figures in Table 2 may be used to determine the number of incremental samples to be taken.

Table 2: Number of incremental samples to be taken depending on the weight of the lot of cereals and cereal products

Lot weight (tonnes)	No of incremental samples
≤ 0.05	3
> 0.05 - ≤ 0.5	5
> 0.5 - ≤ 1	10
> 1 - ≤ 3	20
> 3 - ≤ 10	40
> 10 - ≤ 20	60
> 20 - ≤ 50	100

Sampling procedure for cereals and cereal products for lots >>> 500 tonnes

Number of incremental samples (of about 100 g) to be taken:

100 incremental samples + √metric tonnes

Static Lots

A static lot can be defined as a large mass of cereals/cereal-based product contained either in a large single container such as a wagon, truck or railcar or in many small containers such as sacks or boxes and the cereal/cereal-based product is stationary at the time a sample is selected. Selecting a truly random sample from a static lot can be difficult because all containers in the lot or sublot may not be accessible.

Taking incremental samples from a static lot usually requires the use of probing devices to select product from the lot. The probing devices should be specifically designed for the commodity and type of container.

The probe should (1) be long enough to reach all products, (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 incremental samples 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), as follows:

SF = (LT x IS)/(AS x IP).

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

Dynamic Lots

Representative aggregate samples can be more easily produced when selecting incremental samples from a moving stream of cereals/cereal-based product as the lot is transferred from one location to another. When sampling from a moving stream, take small incremental samples of product from the entire length of the moving stream; composite the incremental samples to obtain an aggregate sample; if the aggregate sample is larger than the required laboratory sample(s), then blend and subdivide the aggregate sample to obtain the desired size laboratory sample(s).

Automatic sampling equipment such as a cross-cut sampler is commercially available with timers that automatically pass a diverter cup through the moving stream at predetermined and uniform intervals. When automatic sampling equipment is not available, a person can be assigned to manually pass a cup through the stream at periodic intervals to collect incremental samples. Whether using automatic or manual methods, incremental samples should be collected and put together at frequent and uniform intervals throughout the entire time the 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 the 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 two to three times the largest dimensions of items in the lot.

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

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

where D is the width of the diverter cup opening (cm), LT is the lot size (kg), T is interval or time between cup movement through the stream (seconds), and V is cup velocity (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 can be computed as a function of S, V, D, and MR.

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

Packaging and Transportation of Samples

Each laboratory sample shall be placed in a clean, inert container offering adequate protection from contamination, sunlight, 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. Samples should be stored in a cool dark place.

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.

SAMPLE PREPARATION

Precautions

Sunlight should be excluded as much as possible during sample preparation, since some mycotoxins may gradually break down under the influence of ultra-violet light. Also, environmental temperature and relative humidity should be controlled and not favour mould growth and deoxynivalenol formation.

Homogenization - Grinding

As the distribution of deoxynivalenol is non-homogeneous, laboratory samples should be completely homogenized by grinding the entire laboratory sample received by the laboratory. Homogenization is a procedure that reduces particle size and disperses the contaminated particles evenly throughout the comminuted laboratory sample.

The laboratory sample should be finely ground and mixed thoroughly using a process that approaches as complete homogenization as possible. Complete homogenization implies that particle size is extremely small and the variability associated with sample preparation approaches zero. After grinding, the grinder should be cleaned to prevent deoxynivalenol cross-contamination.

Test portion

The suggested weight of the test portion taken from the comminuted laboratory sample should be approximately 25 g.

Procedures for selecting the 25 g test portion from the comminuted laboratory sample should be a random process. If mixing occurred during or after the comminution process, the 25 g test portion can be selected from any location throughout the comminuted laboratory sample. Otherwise, the 25 g test portion should be the accumulation of several small portions selected throughout the laboratory sample.

It is suggested that three test portions be selected from each comminuted laboratory sample. The three test portions will be used for enforcement, appeal, and confirmation if needed.