CODEX ALIMENTARIUS COMMISSION

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World Health Organization

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Agenda Item 10(b) **CX/CF 20/14/10 -Part II**

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JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON CONTAMINANTS IN FOODS

14th Session

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PROPOSED SAMPLING PLANS AND PERFORMANCE CRITERIA FOR TOTAL AFLATOXINS IN CERTAIN CEREALS AND CEREAL-BASED PRODUCTS INCLUDING FOODS FOR INFANTS AND YOUNG CHILDREN

(Prepared by the Electronic Working Group chaired by Brazil and co-chaired by India)

Codex members and observers wishing to submit comments at Step 3 on this document should do so as instructed in CL 2020/60/OCS-CF available on the Codex webpage/Circular Letters:
http://www.fao.org/fao-who-codexalimentarius/resources/circular-letters/en/
or on the Codex webpage/CCCF/Related Circular Letters
http://www.fao.org/fao-who-codexalimentarius/committees/committee/en/?committee=CCCF

BACKGROUND

1. CCCF13 agreed to establish an Electronic Working Group (EWG) chaired by Brazil and co-chaired by India to present at its next session proposals for maximum levels (MLs) for total AFs in maize grain destined for further processing, flour, meal, semolina and flakes derived from maize, husked and polished rice (excluding parboiled rice), cereal-based food for infants and young children and sorghum. The Committee further agreed to include sorghum in the list noting that it was a staple food in many parts of the world and that once the work on the MLs for the food categories mentioned above were completed, the proposal of MLs for other cereals and cereal-based products should be considered. There was also agreement that a call for data should be issued on whole wheat flour and parboiled rice to better assess whether these food categories should be added later.[1]

2. Proposals for MLs on total AFs in these food categories can be found in CX/CF 20/14/10-Part I (Appendix I). In addition, comments were requested through CL 2020/23-CF on the opportunity to develop sampling plans and performance criteria for the analysis of total aflatoxins for the relevant food categories and corresponding MLs and if so, whether performance criteria for AFs should consider 70% of total aflatoxins would be AFB1 and the remaining 30% would be distributed equally between AFB2, AFG1 and AFG2 taking into account the issues in CX/CF 20/14/10-Part I (paragrah 6) and the data analysis provided in Appendix II of the same document. Based on the replies provided in CXCF 20/14/10-Add.1, the EWG developed sampling plans as described in Appendix I of this document.

3. This additional task is dependent on time available for the EWG to complete the task entrusted to it by CCCF13 for the consideration of MLs.

4. The sampling plan proposal considered the FAO Sample Tool and the existing sampling plans for fumonisins in maize grain and maize flour and for deoxynivalenol in cereal-based foods for infants and young children in order to harmonize them as extend as possible.

RECOMMENDATIONS

5. In view of the postponement of 14th Session of the Codex Committee on Contaminants in Foods (CCCF14) from 2020 to 2021 due to the pandemic situation, Codex members and observers are kindly invited to consider the sampling plan for total aflatoxins (AFT; AFB1 + AFB2 + AFG1 +AFG2) in cereals grains (maize and sorghum destined for further processing; polished and husked rice); in flour, semolina and flakes derived from maize; and in cereal-based products including foods for infants and young children.

[1] REP19/CF, paras. 146-155, Appendix IX

6. Codex members and observers are invited to consider the sampling plans as contained in Appendix I and provide comments as follows:

   (a) if a sampling plan for total aflatoxins in AFT; AFB1 + AFB2 + AFG1 +AFG2) in cereals grains (maize and sorghum destined for further processing; polished and husked rice); in flour, semolina and flakes derived from maize; and in cereal-based products including foods for infants and young children should be developed;

   (b) the attached proposed draft sampling plan for total aflatoxins in AFT; AFB1 + AFB2 + AFG1 +AFG2) in cereals grains (maize and sorghum destined for further processing; polished and husked rice); in flour, semolina and flakes derived from maize; and in cereal-based products including foods for infants and young children.

APPENDIX I
(For comments)

SAMPLING PLAN FOR TOTAL AFLATOXINS (AFT; AFB1 + AFB2 + AFG1 +AFG2) IN CEREALS GRAINS (maize and sorghum destined for further processing; polished and husked rice); FLOUR, SEMOLINA AND FLAKES DERIVED FROM MAIZE; AND CEREAL-BASED PRODUCTS INCLUDING FOODS FOR INFANTS AND YOUNG CHILDREN

Maize grain destined for further processing

Maximum level	20 µg/kg AFT or 15 µg/kg AFT
Increments	increments of 100 g, depending on the lot weight (≥ 0.5 tons)
Sample preparation	dry grind with a suitable mill (particles smaller than 0.85 mm - 20 mesh)
Laboratory sample weight	≥ 1 kg
Number of laboratory samples	1
Test portion	50 g test portion
Method	HPLC
Decision rule	If the total aflatoxin-sample test result for the laboratory samples is equal or less than 20 µg/kg / 15 µg/kg, accept the lot. Otherwise, reject the lot.

Sorghum grain destined for further processing

Maximum level	10 µg/kg AFT or 8 µg/kg AFT
Increments	increments of 100 g, depending on the lot weight (≥ 0.5 tons)
Sample preparation	dry grind with a suitable mill (particles smaller than 0.85 mm - 20 mesh)
Laboratory sample weight	≥ 1 kg
Number of laboratory samples	1
Test portion	50 g test portion
Method	HPLC
Decision rule	If the total aflatoxin-sample test result for the laboratory samples is equal or less than 10 µg/kg / 8 µg/kg, accept the lot. Otherwise, reject the lot.

Husked rice

Maximum level	20 µg/kg AFT or 15 µg/kg AFT
Increments	increments of 100 g, depending on the lot weight (≥ 0.5 tonnes)
Sample preparation	dry grind with a suitable mill (particles smaller than 0.85 mm - 20 mesh)
Laboratory sample weight	≥ 1 kg
Number of laboratory samples	1
Test portion	50 g test portion
Method	HPLC
Decision rule	If the total aflatoxin-sample test result for the laboratory samples is equal or less than 20 µg/kg / 15 µg/kg, accept the lot. Otherwise, reject the lot.

Polished rice

Maximum level	8 µg/kg AFT or 4 µg/kg AFT
Increments	increments of 100 g, depending on the lot weight (≥ 0.5 tons)
Sample preparation	dry grind with a suitable mill (particles smaller than 0.85 mm - 20 mesh)
Laboratory sample weight	≥ 1 kg
Number of laboratory samples	1
Test portion	50 g test portion
Method	HPLC
Decision rule	If the total aflatoxin-sample test result for the laboratory samples is equal or less than 8 µg/kg / 4 µg/kg, accept the lot. Otherwise, reject the lot.

Flour, meal, semolina and flakes derived from maize

Maximum level	15 µg/kg AFT or 10 µg/kg AFT
Increments	10 x 100 g
Sample preparation	None
Laboratory sample weight	≥ 1 kg
Number of laboratory samples	1
Test portion	25 g test portion
Method	HPLC
Decision rule	If the total aflatoxin-sample test result for the laboratory samples is equal or less than 15 µg/kg / 10 µg/kg, accept the lot. Otherwise, reject the lot.

Cereal-based foods for infants and young children

Maximum level	2 µg/kg AFT or 1 µg/kg AFT
Increments	10 x 100 g
Sample preparation	None
Laboratory sample weight	≥ 1 kg
Number of laboratory samples	1
Test portion	25 g test portion
Method	HPLC
Decision rule	If the total aflatoxin-sample test result for the laboratory samples is equal or less than 2 µg/kg / 1 µg/kg, accept the lot. Otherwise, reject the lot.

DEFINITION

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 - it is defined by an AFT test procedure and an accept/reject level. An AFT test procedure consists of three steps: sample selection, sample preparation and analysis or AFT 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 shelled cereal 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 in such a way to ensure that the laboratory sample is still representative of the sublot sampled.

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 AFT for chemical analysis.

SAMPLING PLAN DESIGN CONSIDERATIONS

Material to be sampled

Each lot of cereal, which is to be examined for AFT, must be sampled separately. Lots larger than 50 tons should be subdivided into sublots to be sampled separately. If a lot is greater than 50 tons, the lot should be subdivided into sublots according to Table 1.

Table 1. Subdivision of maize sublots according to lot weight

Lot weight (t)	Maximum Weight or minimum number of sub lots	Number of incremental sample	Minimum laboratory Sample Weight (kg)
≥ 1500	500 tons	100	1
> 300 and < 1500	3 sublots	100	1
≥ 100 and ≤ 300	100 tons	100	1
≥ 50 and < 100	2 sublots	100	1
< 50	-	3-100*	1

* see table 2

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

Incremental Sample

The suggested minimum weight of the incremental sample should be 100 grams for lots ≥ 0.5 tons.

For lots less than 50 tons, the sampling plan must be used with 3 to 100 incremental samples, depending on the lot weight. For very small lots (≤ 0.5 tons) 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. 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

Lot weight (t)	Number of incremental sample	Minimum Laboratory Sample Weight (kg)
≤ 0.05	3	1
> 0.05 - ≤ 0.5	5	1
> 0.5 - ≤ 1	10	1
> 1 - ≤ 3	20	1
> 3 - ≤ 10	40	1
> 10 - ≤ 20	60	1
> 20 - < 50	100	1

STATIC LOTS

A static lot can be defined as a large mass of shelled cereal 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 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 shelled cereal 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 composited at frequent and uniform intervals throughout the entire time the cereal 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 x LT) / (T x 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 x V) / (D x 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.

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

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

As the distribution of AFT is extremely non-homogeneous, laboratory samples should be 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 AFT cross-contamination.

TEST PORTION

The suggested weight of the test portion taken from the comminuted laboratory sample should be approximately 25 g or 50g, depending on the product analysed.

When analytical method used requests smaller test portions, this can be applied since the performance of the method still fits the purpose and does not impact the uncertainty of the result.

Procedures for selecting the test portion from the comminuted laboratory sample should be a random process. If mixing occurred during or after the comminuting process, the test portion can be selected from any location throughout the comminuted laboratory sample. Otherwise, the 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.

ANALYTICAL METHODS

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. 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 criteria for Total Aflatoxins.

Commodity	**ML** (μg/kg)	**LOD** (μg/kg)	**LOQ** (μg/kg)	**RSDR (%)**	**Minimum applicable range** (μg/kg)	**Recovery**
Maize for further processing	20	≤ 4.0	≤ 8.0	44	11.2 - 28.8	60-115%
Maize for further processing	15	≤ 3.0	≤ 6.0	44	8.4 – 21.6	60-115%
Sorghum for further processing	10	≤ 2.0	≤ 4.0	44	5.6 – 14.4	60-115%
Sorghum for further processing	8	≤ 1.6	≤ 3.2	44	4.48 – 11.52	60 – 115%
Husked rice	20	≤ 4.0	≤ 8.0	44	11.2 - 28.8	60-115%
Husked rice	15	≤ 3.0	≤ 6.0	44	8.4 – 21.6	60-115%
Polished rice	8	≤ 1.6	≤ 3.2	44	4.48 – 11.52	60-115%
Polished rice	4	≤ 0.4	≤ 0.8	44	2.24 – 5.76	40 – 120%
Flour, semolina, meal and flakes derived from maize	15	≤ 3.0	≤ 6.0	44	8.4 – 21.6	60-115%
Flour, semolina, meal and flakes derived from maize	10	≤ 2.0	≤ 4.0	44	5.6 – 14.4	60-115%
Cereal-based foods for infants and young children	2	≤ 0.4	≤ 0.8	44	1.12 – 2.88	40 -120 %
Cereal-based foods for infants and young children	1	≤ 0.2	≤ 0.4	44	0.56 – 1.44	40 – 120%

APPENDIX II

LIST OF PARTICIPANTS
(See CX/CF 20/14/10-Part I)