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SAMPLING PLANS AND NUMERIC PERFORMANCE CRITERIA FOR METHODS OF ANALYSIS FOR TOTAL AFLATOXINS AND OCHRATOXIN A IN CERTAIN SPICES

(Prepared by the Electronic Working Group chaired by India)

BACKGROUND

1. The 17th Session of the Codex Committee on Contaminants in Foods (CCCF17, 2024) recognized the need to develop specific sampling plans and method performance criteria for the analysis of total aflatoxins and ochratoxin A in selected spices, namely nutmeg, dried chilli, and paprika. This initiative responds to the known health risks posed by these mycotoxins and the challenges of ensuring representative sampling in highly variable spice matrices. Given the complexity of developing sampling plans that are fit-for-purpose, the Committee re-established the Electronic Working Group (EWG), chaired by India, to review outstanding issues and further develop the sampling plan. The revised sampling plan would be circulated for comments and consideration by CCCF18.
2. The mandate of the EWG was to finalise the sampling plan (Appendix I), ensuring consistency with Codex guidance and the *General Standard for Contaminants in Food and Feed* (CXS 193-1995), taking into account considerations of commodity value, practicality of implementation, and numeric performance criteria for methods of analysis.¹
3. CCCF17 also agreed to forward the sampling plans to the 47th Session of the Codex Alimentarius Commission (CAC47, 2024) for adoption at Step 5. CAC47 adopted the sampling plans at Step 5 as proposed by the Committee.²

WORK PROCESS

4. The Electronic Working Group (EWG), comprising 17 member countries, conducted its work through two rounds of consultations. Comments on the first draft were received from Indonesia, Thailand, and the United States of America (USA); while the second draft received input from Brazil, Indonesia, and the USA. A summary of the comments submitted during the EWG discussions is tabulated in Appendix II. The list of participants is presented in Appendix III.
5. The Electronic Working Group (EWG) carried out its activities virtually via the EWG online forum. The first draft of the sampling plan was circulated for comments in October 2024, with the comment period extending through December 2024. Following the review of the received comments, a second draft was circulated in January 2025, with a deadline for submitting further comments set for February 2025.
6. Key elements of the EWG's work included:
 - Reviewing analytical literature on aflatoxins in spices.
 - Comparing sampling methods with those used in cereals, nuts, and dried fruits.
 - Evaluating analytical precision and recovery standards.
 - Considering practicality for developing countries and small exporters.
7. The revised draft sampling plan reflects updates based on the discussions and suggestions from member countries, particularly regarding sample weights, analytical precision, and the structure of the sampling tables. Key changes from the previous draft include clarification of subplot structures, improved alignment of incremental and aggregate sample sizes across particle types, and revisions to analytical performance criteria based on Codex and international standards.

KEY POINTS OF DISCUSSION

¹ REP24/CF17, paras. 81-92

² REP14/CAC47, para. 68(ii), Appendix III

8. **Sample Weight for Large Particle Spices:** Indonesia proposed increasing the incremental sample weight from 100 g to 200 g to improve representativeness. However, CCCF17 had previously agreed on a minimal weight considering the high value of spices. The proposal was not adopted.
9. **Syntax Corrections in Tables:** Editorial corrections to avoid overlapping definitions in lot weight ranges were accepted (e.g., changing ">10 – ≤15" to ">10 – <15").
10. **Powdered Spices Sampling Plan:** Due to heterogeneity and practical concerns, multiple values for incremental sample size ([40 g], [80 g]) and aggregate sample weight ([0.1 – 4.0 kg]) remain under discussion and are enclosed in [square brackets].
11. **Analytical Method Performance:** Proposed changes to method precision (≤20%) and recovery (70–120%) were not adopted due to a lack of alignment with internationally accepted ranges. Current values (≤44% precision, 60–115% recovery) reflect realistic expectations at low contamination levels, consistent with EU regulations and Codex practice.
12. **Reformatting of Tables:** Table headers were updated for clarity (e.g., explicitly stating commodity type). The columns for "weight or number of sublots" were retained to preserve technical completeness despite some requests to simplify.
13. **AFB1 as Separate Analyte:** A separate maximum level (ML) of 15 µg/kg for AFB1 was added in Table 7, reflecting recognition of its higher toxicity and harmonisation with international practice.

CONCLUSIONS

14. The EWG successfully developed a harmonised sampling plan for spices of various particle sizes and states (whole, crushed, powdered). The EWG has significantly improved the structure, clarity, and scientific soundness of the sampling plan. The updated draft:
 - Differentiates sampling approaches for spices by particle size.
 - Incorporates detailed tables to guide incremental and aggregate sampling.
 - Provides method performance criteria based on international norms.
15. Unresolved issues related to powdered spices sampling (incremental sample sizes and aggregate sample weights) remain bracketed for further discussion.
16. It was decided to
 - Maintain a 100 g incremental sample weight.
 - Keep precision at ≤44%, in line with established international practices.
 - Retain 60–115% recovery for most analytical ranges.
 - Consider establishing a separate ML for AFB1, subject to broader CCCF consensus.

RECOMMENDATIONS

17. CCCF is invited to consider the following:

Sampling plans

- (i) The sampling plan structure, subdivision logic, and sample size criteria as presented in Appendix I, including those provisions that remained unsolved, shown in square brackets, and the numeric performance criteria with a view to their finalisation by CCCF18.
- (ii) Reject the proposed change to ≤20% precision and mandatory 200 g incremental samples, as these lack scientific and operational feasibility.
- (iii) Correct overlapping notations in tables (e.g., ">10–≤15" to ">10–<15") to avoid interpretation errors.

Numeric performance criteria for methods of analysis

- (iv) Retain the current method performance criteria for:
 - Precision at ≤44%.
 - Recovery at 60–115%.

Other matters

- (v) Consider the adoption of a separate ML for AFB1 (15 µg/kg) in future discussions, given its toxicological significance and alignment with international precedents.

APPENDIX I

**SAMPLING PLAN FOR TOTAL AFLATOXINS AND OCHRATOXIN A IN
NUTMEG, DRIED CHILLI AND PAPRIKA**

(For comments at Step 6)

A) Spices with large particle size (Whole nutmeg, whole dried chilli and whole paprika)

In case of large lots and on condition that the subplot can be separated physically, each lot shall be subdivided into sublots following Table 1. 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 subplot may exceed the mentioned weight in Table 1 by a maximum of 20 %.

**Table 1: Subdivision of Spices sublots according to lot weight
– Whole nutmeg, whole dried chilli and whole paprika -**

Lot weight (tonne)	Weight or number of sublots	No incremental samples	Aggregate sample weight (kg)
≥ 500	100 tonnes	100	10
> 125 and < 500	5 sublots	100	10
≥ 25 and ≤ 125	25 tonnes	100	10
< 15	—	10-100 (*)	10
(*) Depending on the lot weight — see Table 2.			

Each sub-lot shall be sampled separately. The number of incremental samples of 100 g 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 2 shall be used to determine the number of incremental samples to be taken and the subsequent division of the aggregate sample.

**Table 2: Number of incremental samples to be taken according to lot weight
– Whole nutmeg, whole dried chilli and whole paprika -**

Lot weight (tonnes)	No of incremental samples	Aggregate sample Weight (kg)
≤ 0.1	10	1
> 0.1 – ≤ 0.2	15	1.5
> 0.2 – ≤ 0.5	20	2
> 0.5 – ≤ 1.0	30	3
> 1.0 – ≤ 2.0	40	4
> 2.0 – ≤ 5.0	60	6
> 5.0 – ≤ 10.0	80	8
> 10.0 – <15.0	100	10

The accept/reject level is a level usually equal to the Codex maximum level.

B) Spices with small particle size (crushed/cracked/broken/flakes of nutmeg, dried chilli and paprika)

In the case of large lots and on condition that the subplot can be separated physically, each lot shall be subdivided into sublots following Table 3. 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 subplot may exceed the mentioned weight in Table 3 by a maximum of 20 %.

Table 3 Subdivision of spices sublots according to lot weight
- crushed/cracked/broken/flakes of nutmeg, dried chilli and paprika -

lot weight (tonnes)	Weight or number of sublots	Number of incremental samples	Aggregate sample Weight (kg)
≥ 25	25 tonnes	100	10
< 25	—	5-100 (*)	0.5-10
(*) Depending on the lot weight — see Table 4			

Each subplot shall be sampled separately. The number of incremental samples of 100g to be taken depends on the lot weight, with a minimum of 5 and a maximum of 100. resulting in an aggregate sample of 0.5 to 10 kg. Table 4 can be used to determine the number of incremental samples to be taken from lots of various sizes.

Table 4 Number of incremental samples to be taken according to lot weight
- crushed/cracked/broken/flakes of nutmeg, dried chilli and paprika -

Lot weight (tonnes)	Number of incremental samples	Aggregate sample weight (kg)
≤ 0.01	5	0.5
> 0.01-≤ 0.1	10	1
> 0.1-≤ 0.2	15	1.5
> 0.2-≤ 0,5	20	2
> 0,5-≤ 1.0	30	3
> 1.0-≤ 2.0	40	4
> 2.0-≤ 5.0	60	6
> 5.0-≤ 10.0	80	8
> 10.0-≤ 25.0	100	10

C) Powdered spices (obtained by grinding nutmeg, dried chilli and paprika) In the case of large lots and on condition that the subplot can be separated physically, each lot shall be subdivided into sublots following Table 5. 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 subplot may exceed the mentioned weight in Table 5 by a maximum of 20 %.

**Table 5 Subdivision of spices sublots according to lot weight
- Powdered spices (nutmeg, dried chilli and paprika) -**

Lot weight (tonnes)	Weight or number of sublots	Number of incremental samples	Aggregate sample Weight (kg)
≥ 25	25 tonnes	50	2 [4]
< 25	—	3 – 50 (*)	0.1 – 2.0 [0.24 – 4.0]
(*) Depending on the lot weight — see Table 6			

Each subplot shall be sampled separately. The number of incremental samples [40 g] [80 g] to be taken depends on the lot weight with a minimum of 3 and maximum 50 incremental samples. Table 6 can be used to determine the number of incremental samples to be taken from lots of various sizes.

Table 6 Number of incremental samples of Powdered Spices to be taken depending on the weight of the lot

Lot weight (tonnes)	Minimum number of incremental samples	Minimum aggregate sample weight (kg)
≤ 0.1	3	0.1 [0.24]
> 0.1 - ≤ 0.5	10	0.4 [0.8]
> 0.5 - ≤ 5.0	25	1.0 [2]
> 5.0 - ≤ 10.0	35	1.4 [2.8]
> 10.0 - ≤ 15.0	50	2.0 [4]

Method performance criteria

Table 7: Method performance criteria for the analysis of total aflatoxins and ochratoxins A in spices

Commodity	Analyte	ML (µg/kg)	LOD (µg/kg)	LOQ (µg/kg)	Precision (%)	Minimal applicable range (µg/kg)	Recovery (%)
Chilli pepper, Nutmeg	AFT B1+B2+G1+G2	20	≤4	≤8	≤44	11.2-28.8	60-115
	AFB1	15	≤1	≤2	≤44	2.8-7.2	40-120
	AFB2	-	≤1	≤2	≤44	2.8-7.2	40-120
	AFG1	-	≤1	≤2	≤44	2.8-7.2	40-120
	AFG2	-	≤1	≤2	≤44	2.8-7.2	40-120
Chilli pepper, Paprika, nutmeg	OTA	20	≤4	≤8	≤44	11.2-28.8	60-115

APPENDIX II
Compilation of comments & Justification
(For information)

S.No	Country	Section	Comment	Justification by members	Action/Remarks
1.	Indonesia	Section A Para 2	Indonesia proposes that the weight for each incremental sample of spices with a larger particle size is 200g.	The weight for each incremental sample of 100 g is not representative of the sampled lot/batch. It might not adequately represent the quantity and variability of samples at each sampling point.	Under REP24/CF17 Pg No11 Sr.No 87, its decided considering the high value of spices, minimal weight to be considered.
2	Indonesia	Table 2	The number of incremental samples of 200 g instead of the proposed 100g.	In par with the previous comment.	Under REP24/CF17 Pg No11 Sr.No 87, its decided considering the high value of spices, minimal weight to be considered.
3	Indonesia	Table 2	Under Lot weight (tonnes) $> 10.0 - \leq 15.0$ should be changed to $> 10.0 - < 15.0$	Considering overlapping from Table 1.	Accepted.
4	Indonesia	Table 4	Under Lot weight (tonnes) $> 10.0 - \leq 15.0$ should be changed to $> 10.0 - < 15.0$	Considering overlapping from Table 1.	Accepted.
5	Indonesia	Table 5	Under column Aggregate sample Weight (kg) – include [4] for ≥ 15 and include [0.24 – 4.0] for < 15	Incremental sample size: [40 g] [80 g] were mentioned and weight under debate hence corresponding weight included.	Accepted.
6	Indonesia	Table 7	Precision Change ($\leq 20\%$ instead of $\leq 44\%$)		$\leq 20\%$ precision is very strict. Standard methods like HPLC-FLD, LC-MS/MS (most common toxin testing methods) usually achieve $\sim 20\%$ precision at higher concentrations (near $20 \mu\text{g/kg}$) BUT at very low concentrations (e.g., $1-5 \mu\text{g/kg}$), precision naturally worsens (more than 20%) EU regulations for mycotoxins often allow up to $40-45\%$

S.No	Country	Section	Comment	Justification by members	Action/Remarks
					precision at low levels (example: EU Regulation 401/2006 allows 44% precision at low levels). For low-level contamination (which is the case here), expecting $\leq 20\%$ precision is not practically achievable with many standard testing methods. Scientifically, $\leq 44\%$ is more realistic and widely accepted internationally.
7	Indonesia	Table 7	Recovery Change (70–120% instead of 60–115%)		Current standards (like Codex, EU) generally accept: 60–120% recovery for low concentrations ($< 10 \mu\text{g/kg}$). 70–110% or 70–120% for higher concentrations. Indonesia's suggestion (70–120%) is acceptable if applied only for concentrations around $20 \mu\text{g/kg}$. But at very low toxin levels (near $1\text{--}5 \mu\text{g/kg}$), 60–115% recovery is already considered good enough. Raising the recovery window slightly (to 70–120%) may be okay if testing concentrations are higher, but for very low levels, it might unfairly reject otherwise good methods.
8	Indonesia	Table 7	New ML for AFB1 ($15 \mu\text{g/kg}$)	EU regulations already set AFB1 limits separately, e.g.: $5 \mu\text{g/kg}$ for AFB1 in many spices (much stricter than $15 \mu\text{g/kg}$!) Many countries recognize AFB1 as highly carcinogenic. Having a separate ML for AFB1 at $15 \mu\text{g/kg}$ is scientifically reasonable and actually aligns better with consumer safety goals.	Setting an ML for AFB1 separately (at $15 \mu\text{g/kg}$) is scientifically justified. Accepted.
9	Brazil	Table 1, 2, 3, 4, 5, 6 Heading	The title should reflect that it is applicable only.		Accepted.
10	Brazil	Table 1, 3	Suggested		Weight and subplot given

S.No	Country	Section	Comment	Justification by members	Action/Remarks
		& 5	"weight or number of subplot" column be limited do number of sublots for better understanding.		together, hence cannot be removed.
11	Brazil	Table 6	"Weight of incremental samples of [0.24 kg to 4kg], resulting in a aggregate sample of [0.1 to 2.0 kg].		The incremental samples are from 3 to 50 and not 0.24 kg to 4 kg. Clarity required.
12	Thailand	Table 7	Previously established values of precision and recovery need to be kept as such.	As outlined in Table 7 of REP24/CF1–Appendix IV, are in line with the method performance of total aflatoxins in cereals, particularly regarding husked rice with the same ML of 20 µg/kg in the General Standard for Contaminants and Toxins in Food and Feed (CXS 193-1995). This is endorsed by the CCMAS42.	Accepted.
13	USA	Tables 1, 3 & 5	This column is not necessary here, as well as in Tables 3 and 5.		Accepted.
14	USA	Tables 1 to 6 Headings	Headings should define the commodity.		Accepted.
15	USA	Tables 1, 3, 5 & 6	Suggesting a value of 25 instead of 15 under the lot weight.	Can't have 25 tonne subplot from 15-24.9 tonnes. Should this be >= 25?	Accepted.

**APPENDIX III
LIST OF PARTICIPANTS**

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