CODEX ALIMENTARIUS COMMISSION

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Food and Agriculture Organization of the United Nations

World Health Organization

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REP23/MAS Corrigendum*

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX ALIMENTARIUS COMMISSION

46th Session

Rome, Italy 27 November – 2 December 2023

REPORT OF THE 42nd SESSION OF THE CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

13 – 16 June 2023 physical plenary meeting and

20 June 2023 virtual report adoption

*** See Appendix II***

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Responsible Party	Purpose	Text / Topic	Code / Reference	Step	Para(s)
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Brazil CCMAS43	Drafting / Discussion	Discussion paper to consider harmonization of names and format for principles	CXS 234-1999	-	50, 57(vi)
Chile, Brazil CCMAS43	Drafting / Discussion	Discussion paper to explore the best approach for the placement of nitrogen conversion factors	CXS 234-1999	-	53, 57(vi)
EWG (Uruguay, Brazil) / CCMAS43	Drafting / Discussion	Discussion paper on Listing of Type IV methods in CXS 234 when a Type I method is listed for the same commodity and provision.	CXS 234-1999	-	56, 57(vii)
EWG (Canada) / PWG on	Review / Update	Cereals, pulses and legumes workable package	CXS 234-1999	-	63

Responsible Party	Purpose	Text / Topic	Code / Reference	Step	Para(s)
endorsement CCMAS43					
EWG (Norway) / PWG on endorsement / CCMAS43	Review / Update	Fish and Fishery products	CXS 234 – 1999	-	66(ii)(a)
EWG (Germany) / PWG on endorsement / CCMAS43	Review / Update	Fruit juices	CXS 234 - 1999	-	66(ii)(b)
EWG (New Zealand / Germany)/ Members/ CCMAS43	Drafting / Discussion	Information document (e-book with sampling plans applications)	CXG 50-2004	-	81(ii)

LIST OF ABBREVIATIONS

AOAC	AOAC International (formerly known as Association of Official Agricultural Chemists)
AOCS	American Oil Chemists' Society
CAC	Codex Alimentarius Commission
CCAFRICA	FAO/WHO Coordinating Committee for Africa
CCASIA	FAO/WHO Coordinating Committee for Asia
CCNASWP	FAO/WHO Coordinating Committee for North America and South West Pacific
CCCF	Committee on Contaminants in Foods
CCFA	Committee on Food Additives
CCFH	Committee on Food Hygiene
CCFL	Committee on Food Labelling
CCFO	Committee on Fats and Oils
CCMAS	Committee on Methods of Analysis and Sampling
CCNFSDU	Committee on Nutrition and Foods for Special Dietary Uses
CCSCH	Committee on Spices and Culinary Herbs
CCEXEC	Executive Committee of the Codex Alimentarius Commission
CLs	Circular Letters
CRD	Conference room document
CRM	Certified Reference Material
DNA	Deoxyribonucleic acid
ELISA	Enzyme-linked immunosorbent assay
EU	European Union
EWG	Electronic working group
FAO	Food and Agriculture Organization of the United Nations
HPTLC	High performance thin layer chromatography
IAM	Interagency Meeting
IDF	International Dairy Federation
ISO	International Organization for Standardization
LOD	Limit of Determination
LOQ	Limit of Quantification
NFCSO	National Food Chain Safety Office (Hungary)
MCM	Monte Carlo Method
ML	Maximum level
MU	Measurement uncertainty
PWG	Physical working group
REU	Regional Office for Europe and Central Asia
SDO	Standards development organisations
USPC	United States Pharmacopeial Convention
WG	Working group
WHO	World Health Organization

LIST OF CRDS

CRD No.	Agenda Item	Submitted by
01	Division of Competence	EU (Division of Competence between EU and its Member States)
02	2, 3, 4.1, 4.2 and 4.3	Report of PWG on endorsement
03	6	Revision of the general guidelines on sampling prepared by EWG
04	5	Revised draft information document on Measurement of uncertainty
05	3	Regional coordinator on behalf of CCNASWP
06	3	Information submitted by AOAC, ISO and IDF
07	3	IDF and ISO
08	4.1	EWG of Fats and Oils workable package
09	2	Chile, Uruguay
10	3	Chile, EU, Ghana, Uruguay
11	4.1	Chile, Ghana
12	4.2	Chile, Colombia, Ghana, Nigeria, Peru, Uruguay
13	4.3	Chile, Ghana, Nigeria, Peru, Uruguay
14	3	Argentina, Brazil, Uruguay
15	5	Chile, Ghana, Nigeria, Peru, Uruguay
16	6	Ghana, Nigeria, Peru, Thailand, Uruguay
17	7	Chile, Ghana, Uruguay
18	3, 5, and 6	Senegal
19	3	IACST
20	1, 2, 3, 4.1, 4.2, 4.3, 5, 6, and 7	Kenya
21	4.1, 4.2, 5, 6, and 7	Uganda
22	6	Eurachem
23	4.2, 4.3	Thailand
24	8	The report of IAM
25	6	The report of In-session WG on revision of the general guidelines on sampling

INTRODUCTION

1. The Codex Committee on Methods of Analysis and Sampling (CCMAS) held its 42nd Session from 13 – 16 June 2023 and virtual adoption of the report on 20 June 2023, at the kind invitation of the Government of Hungary. The Session was chaired by Dr Attila Nagy, Director, National Food Chain Safety Office (NFCSO) and Dr Zsuzsa Farkas, Food Chain Data Scientist, Digital Food Institute acted as the Vice-Chairperson. The Session was attended by 49 Member Countries and 1 Member Organization, 13 Observer Organizations and Palestine. A list of participants is given in Appendix I.

OPENING OF THE SESSION

2. The Session was opened by Dr Márton Nobilis, the State Secretary of the Ministry of Agriculture of Hungary who welcomed delegates and highlighted the importance of the Committee's continuing work on harmonized and reliable analytical methods and sampling which make for improved laboratory systems and ensure better food safety. Mr Nabil Gangi, Regional Office for Europe and Central Asia (REU) of the Food and Agriculture Organization of the United Nations (FAO), the chairperson on behalf of the World Health Organization (WHO), and Mr Steve Wearne, the Chairperson of Codex Alimentarius Commission also addressed the Committee.

Division of Competence

3. CCMAS noted the division of competence between the European Union and its Member States, according to paragraph 5, Rule II of the Rules of Procedure of the Codex Alimentarius Commission.

ADOPTION OF THE AGENDA (Agenda Item 1)[1]

4. CCMAS adopted the Provisional Agenda as the agenda for the session.

MATTERS REFERRED TO THE COMMITTEE BY THE CODEX ALIMENTARIUS COMMISSION AND OTHER SUBSIDIARY BODIES (Agenda Item 2)[2]

5. CCMAS:

- noted the matters for information referred by the Codex Alimentarius Commission (CAC) the Executive Committee of the Codex Alimentarius Commission (CCEXEC) and the matters for action arising from CCFA, CCFO, CCCF, CCFH and CCFL would be considered under Agenda Item 3 (Endorsement of methods of analysis and sampling); and
- the ongoing work on sampling plans for methylmercury in fish in CCCF;
- encouraged Members and Observers, on the occasion of the 60th anniversary of Codex, to plan and implement activities to build awareness of Codex and to engage high level political support for Codex work and to consider the implementation of a regional event to mark the 60th anniversary;
- encouraged Members and Observers to actively engage in opportunities to contribute to the discussions in CCEXEC and CAC (i.e. the operationalization of the Statements of principle concerning the role of science in the Codex decision-making process and the extent to which other factors are taken into account; the future of Codex; new food sources and production systems, and monitoring the use of Codex standards) by providing replies to relevant Circular Letters (CLs).

6. The Codex Secretariat also presented the innovations regarding the publication of Codex texts, the new Codex website and how these initiatives were linked to Goal three of the Codex Strategic Plan 2020-2025, "Increase impact through the recognition and use of Codex standards".

7. Of specific relevance to CCMAS will be the opportunities on the new website to associate related documents to texts that CCMAS develops and the possibility to collaborate on the development of a database for *Recommended Methods of Analysis and Sampling* (CXS 234-1999) and the e-book applications for the *General Guidelines on Sampling* (CXG 50-2004). It was recommended that ample discussion take place on these digital innovations to ensure they meet the needs of all users.

8. The Codex Secretariat proposed to carry out a scoping exercise on the development of a database for CXS234, with interested delegations from the Committee. This would ensure from the outset that the project took into consideration the end user experience as well as the needs of CCMAS and of the wider Codex community.

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1 CX/MAS 23/42/1
2 CX/MAS 23/42/2

ENDORSEMENT OF METHODS OF ANALYSIS AND SAMPLING PLANS FOR PROVISIONS IN CODEX STANDARDS (Agenda Item 3)[3]

9. CCMAS considered the recommendations on methods of analysis and sampling proposed for endorsement and other related matters as presented in CRD02.

10. CCMAS made the following decisions which are also presented in Appendix II as follows.

Codex Committee on Contaminants in Foods (CCCF15)

Review of methods of analysis for contaminants: Performance criteria for lead and cadmium

11. CCMAS agreed:

- to the numeric performance criteria (Appendix II, Part A) and as a consequence:
    - that the *General Methods of Analysis for Contaminants* (CXS 228-2001) and methods in CXS 234 for lead and cadmium for the commodities covered by the performance criteria should be revoked (Appendix I, Table II of CRD02);
- to continue to review the methods from Appendix I, Table II in CRD02 and other methods to identify examples of available methods that meet the criteria; and
- to request the Codex Secretariat to issue a CL to request examples of methods and information of these methods that could meet the numeric performance criteria for review by the PWG on endorsement and CCMAS43.

Codex Committee on Food Hygiene (CCFH53)

Review of the methods of analysis for irradiated foods in the *General Standard for Methods for the Detection of Irradiated Foods* (CXS 231-2001) and their incorporation into the CXS 234

12. CCMAS agreed:

- that the methods listed in CXS 231 not be endorsed at this time due to insufficient information available on the methods and the application and as a consequence to maintain the methods in CXS 231;
- to gather additional information on the methods through a CL; and
- that the information and methods be submitted for additional review by the PWG on endorsement and CCMAS43.

Codex Committee on Food Additives (CCFA53)

Testing methods related to nitrates and nitrites

13. CCMAS noted that the Committee needed further consideration to provide a reply to CCFA and agreed to establish an electronic Working Group (EWG) chaired by the United States of America (USA, working in English to:

- establish numeric performance criteria for the determination of nitrate and nitrite ions in the food matrices listed in CX/FA 21/52/7 Appendix 5, Annex 2;
- review the methods in CX/FA 21/52/7 Appendix 5, Annex 1 and determine if these methods meet the numeric performance criteria established for the matrices in CX/FA 21/52/7 Appendix 5, Annex 2;
- discuss if the methods determine both nitrate and nitrite ions and if so, whether the methods detect each ion separately or only in combination; and
- discuss if the different determination schemes (i.e. separate or combined) could have an impact on the precision and accuracy of the methods.

14. CCMAS agreed that the report and recommendations of the EWG would be considered at CCMAS43.

Codex Committee on Food Labelling (CCFL47)

Food allergen labelling – precautionary allergen labelling: methods of analysis and sampling

15. CCMAS noted that it was not ready to provide a reply to CCFL at this time and agreed to establish an EWG chaired by the USA and co-chaired by the United Kingdom (UK) working in English, to develop a discussion paper which would discuss best practices for the selection of validated analytical methods, and for the validation of such methods.

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[3] CX/MAS 23/42/2; CX/MAS 23/42/2-Add.1; CX/MAS 23/42/3; CX/MAS 23/42/3-Add.1

16. The discussion paper should consider the following for the allergens listed in Table 11 of the FAO/WHO report *"Risk Assessment of Food Allergens Part 2: Review and Establish Threshold Levels in Foods for the Priority Allergens"*

- Define standardized and harmonized terminology and definitions for allergen testing methods.
- Currently available test methods and validation status for the priority allergenic proteins listed in CX/FL 23/47/5 Appendix I and noting the validated scope (food matrices, processed food) of these methods.
- Required information for method evaluation and validation, including antibodies used (if ELISA), cross-reactivity, assay applicability, selectivity, stability (ruggedness), calibration procedures, sensitivity, range of quantification, LOD/LOQ, accuracy/trueness, extraction efficiency, precision, robustness, applicability, recovery and practicability, and whether it reports total protein. Validation requirements for the testing of allergenic proteins in foods including accuracy/trueness, extraction efficiency, precision, robustness, applicability, recovery and practicability.
- Confirmatory methods for cases of potential analytical cross-reactivity and examples of such possibly including second ELISA confirmation, DNA based detection, and/or mass spectrometry techniques.
- Reference to other 'best practice' guidance documents, include SDO validation procedures and relevant Codex texts.

17. CCMAS agreed that the EWG would not address the question on sampling plans and noted that sampling plans are covered by the *General Guidelines on Sampling* (CXG 50-2004).

18. An observer noted that the definition for food allergen agreed by CCFL47 covered also other specific immune-mediated reactions, which is coeliac disease and that coeliac disease as defined is a chronic immune-mediated intestinal disease in genetically predisposed individuals induced by exposure to dietary gluten proteins that come from wheat, and all *Triticum* species, rye, barley and triticale and that the *Standard for Foods for Special Dietary Use for Persons Intolerant to Gluten* (CXS 118-1979) addresses this and requested that this Standard should be taken into account when addressing the request from CCFL.

Codex Committee on Nutrition and Foods for Special Dietary Uses (CCNFSDU43)

Methods of analysis for provisions in the Standard for Follow-up Formula (CXS 156-1987)

19. CCMAS noted that the request from CCNFSDU (CX/MAS 23/42/3, para. 1) had not been addressed by the PWG.

20. The Codex Secretariat clarified that the request from CCNFSDU follows the revision of the *Standard for Follow-up Formula* (CXS 156-1987) which comprises two parts addressing (i) follow-up formula for older infants and (ii) product for young children. The current CXS 234 contained methods of analysis for certain provisions for follow-up formula and CCMAS should consider whether these methods would also be applicable for product for young children. The revised Standard developed by CCNFSDU has been sent for final adoption by CAC46 and CCMAS could consider the request once the Standard has been adopted.

21. CCMAS agreed to consider this matter at its next session.

Standard for Infant Formula and Formulas for Special Medical Purposes Intended for Infants (CXS 72-1981) methods for vitamin B12, total amino acids (excluding taurine and tryptophan) and tryptophan

22. CCMAS endorsed the methods as proposed (Appendix III, CRD02).

Codex Committee on Spices and Culinary Herbs (CCSCH5)

23. CCMAS:

- did not endorse the methods proposed by CCSCH and agreed to return them for further consideration by CCSCH; and
- requested CCSCH to reply to the following questions to assist CCMAS in the endorsement of the methods as follows:

Standard for dried roots, rhizomes, and bulbs - dried or dehydrated ginger (CXS 343 – 2021); the *Standard for dried floral parts - cloves* (CXS 344-2021) and *Standard for dried leaves – dried basil* (CXS 345-2021)

1. ISO 927 is identified as a Type I method for "whole dead insects", but as a Type IV for 'live insects'. Is there a reason for this difference in typing?
2. MPM-V8 is listed as a Type IV for 'mammalian / other excreta', however ISO 927 appears to capture this category and is identified as a Type I at other parts of the table. Is there a reason for selecting a Type IV for this provision?

Standard for dried floral parts: saffron (CXS 351-2021)

1. The taste strength, aroma strength, colouring strength provisions use the ISO 3632-2 and are listed as Type IV. As this ISO standard is specific to saffron, is there a reason it is listed as a Type IV and not a Type I?

Standard for dried or dehydrated chilli pepper and paprika (CXS 353-2022)

1. For the provision 'live insects' there are two methods listed and both identified as Type I. Are these methods identical? If not, one must be endorsed as Type I method and the other removed.

Draft Standard for spices derived from dried fruits and berries (Part A – allspice, juniper berry and star anise)

1. There are Type I and Type IV methods listed for the provisions "whole dead insects" and "insect fragments". While listing both a Type I and Type IV is allowed, there should be a compelling reason for the listing. Would it be possible to explain the reasoning for this request?
2. There are parenthetical comments in the provision for 'filth' and 'light filth', which says *list all the filth here – for example – mammalian excreta?.* It is unclear if this is text should have been removed.

Comparison between different CCSCH standards

1. In the *Standard for dried roots, rhizomes and bulbs - dried or dehydrated ginger* (CXS 343-2021) ISO 927 is a Type IV for 'mammalian / other excreta', but in the *Standard for dried seeds – nutmeg* (CXS 352-2022) ISO 927 is listed as a Type I for this same provision. Is there a reason for the different typing of the same method for the same provision?
2. In some standards the provision is listed as 'mould visible' and in others it is listed as 'visible mould', is there a significance to this difference or could a single name for the provision be used consistently across standards.
3. Across standards there are some differences in provision groups. One example, in the draft Standard for dried small cardamom the provision is 'whole insect live / dead', while in the *Standard for dried roots, rhizomes and bulbs- dried or dehydrated ginger* (CXS 343-2021), the provisions are listed separately as 'whole dead insects' and 'live insect'. Are these intentional?

24. To a question whether there was a set of definitions / terminologies that could assist CCMAS when the methods are reviewed when selecting a series of methods for foreign and extraneous matters for endorsement, the Codex Secretariat clarified that CCSCH had developed a glossary of terms for their internal use available in CX/SCH 17/3/10.

25. CCMAS agreed to request CCSCH to confirm the availability of this glossary.

FAO/WHO Coordinating Committee for Africa (CCAFRICA24)

Methods of analysis and sampling for provisions in the *Standard for Dried Meat* (CXS 350-2022)

26. CCMAS agreed:

- to endorse the methods of analysis (Appendix II, Part 1);
- to request CCCAFRICA whether the removal of AOAC 935.47 and AOAC 939.09b for determination of chloride was deliberate or if the methods should have been retained as Type III? CCMAS41 had requested CCAFRICA to identify one Type II method from the multiple methods submitted for the determination of chloride and to retain the rest of the methods as Type III[4]; and
- to not endorse the sampling plan and to request CCAFRICA to develop a sampling plan in accordance with the revised *General Guidelines on Sampling* (CXG 50-2005).

FAO/WHO COORDINATING COMMITTEE FOR ASIA (CCASIA22)

Methods of analysis and sampling for provisions in the draft Regional Standard for Soybean Products Fermented with *Bacillus* species

27. CCMAS:

- endorsed the methods of analysis for provisions (Appendix II, Part 1); and
- did not endorse the sampling plan and agreed to request CCASIA to develop a sampling plan for the regional *Standard for Soybean Products Fermented with Bacillus species* in accordance with the revised *General Guidelines on Sampling* (CXG 50-2004) noting the clarification from the Codex Secretariat that lack of sampling plans in commodity standards would not impede the adoption or publication of the standards.

[4] REP21/MAS, para 12ii

The Regional Standard for Cooked Rice Wrapped in Plant Leaves

28. CCMAS:

- endorsed the methods of analysis for determination of peroxide value as Type IV (Appendix II, Part 1); and
- noted that there was no validation data for the oil extraction step from rice establishing the performance (e.g. recovery, precision) and agreed to request CCASIA to undertake validation studies of the extraction method for the rice extraction step to establish the performance (e.g. recovery, precision) of the extraction procedure and to provide the data to CCMAS for review and re-consideration of the Typing.

FAO/WHO Coordinating Committee for North America and South West Pacific (CCNASWP16)

Methods of analysis for provisions in the draft Regional Standard for Fermented Noni Fruit Juice

29. CCMAS:

- endorsed the methods of analysis for scopoletin and for deacetylasperulosidic acid as Type IV (Appendix II, Part 1); and
- endorsed AOAC 983.17 / EN 12143 / IFUMA 8 / ISO 2173 for brix value (soluble solids) as Type IV as they were determined to be identical at CCMAS41 and to wait for the completion of the IFU-led study to reconsider the typing of the method.[5]

Revised SOP for the identification of kavalactones and flavokavains in fresh and dried kava products by HPTLC in the Regional *Standard for Kava Products for Use as a Beverage when Mixed with Water* (CXS 33R-2020)

30. CCMAS:

- did not endorse the SOP due to the lack of description of how the final determination is to be made; and
- requested CCNASWP to provide further edits to address the lack of instruction on the final determination steps and assessment against a specification.

Codex Committee on Contaminants in Foods (CCCF16)

Sampling plans provisions in the *General Standard for Contaminants and Toxins in Food and Feed* (CXS 193-1995)

31. CCMAS:

- endorsed the sampling plan (see Appendix II, Part 1: Sampling plans for total aflatoxins in certain cereals and cereal-based products including foods for infants and young children); and
- requested CCCF to evaluate the sampling plans in CXS 193, including the one just endorsed to determine if the plans were still within the revised *General Guidelines on Sampling* (CXG 50-2004).

Performance criteria – sum of components

32. CCMAS noted that the PWG had agreed with the sum of components approach, but that the footnote was removed since it created a set of multiple numeric criteria at each ML. Instead, a 1:1:1:1 ratio of isomers was used as a basis to calculate a single set of numeric criteria using the sum of components approach, since this was implied by one option provided in the original footnote.

33. CCMAS noted that the Sum of components information document (and the guidance in the Procedural Manual) does allow for the sum of components to be addressed on a case-by-case basis and the Information Document provides examples. A request was made to update the Information Document to reflect this new example.

34. CCMAS:

- endorsed the revised numeric performance criteria proposed by the PWG (Appendix II, Part 1);
- requested the Codex Secretariat to issue a CL to request information on example methods that meet the numeric performance criteria;
- agreed to inform CCCF of the rationale for the revised numeric performance criteria and to recommend that CCCF should provide numeric performance criteria for aflatoxin methods utilizing the sum of components concept for all relevant commodities to replace the existing numeric criteria in CXS 193;

[5] See REP21/MAS, para 13 for information on the IFU-led study.

- agreed to update the Information Document: Sum of components by the addition of the following text to the end of the document:

  *"if the components included in the ML definition are not present in constant ratios and where the inclusion of weighting factors of the individual components results in LOD/LOQ values or minimum applicable range that cannot be validated, ML/n should be used to determine the criterion for LOD (e.g. 1/5*ML/n) and for LOQ (e.g. 2/5*ML/n) or for the minimum applicable range (e.g. ML/n±2S_R), with n being the number of components included in the ML definition".*

Other proposals

Method of analysis for determination of moisture content in dried milk

35. CCMAS recalled that CCMAS41 could not reach consensus on the method ISO 5537 | IDF 26 for determination of moisture content in dried milk and that this matter would be further considered by CCMAS42 and that performance data to evaluate replacement of a Type I method listed in CXS234 would be necessary.[6]

36. CCMAS noted that an alternative proposal had been submitted by Uruguay, Argentina and Brazil together with performance data for consideration by the PWG (CX/MAS 23/42/3, Appendix II Annex 2). The proposal was for CCMAS to endorse the method described in CX/MAS 23/42/3 Appendix II Annex 2 as Type I for the determination of moisture in blend of skimmed milk and vegetable fat in powdered form, reduced fat blend of skimmed milk powder and vegetable fat in powdered form, dairy permeate powders, milk powders and cream powders and whey powders.

37. CCMAS noted that the PWG could not reach consensus on this issue, but that a proposal had been made in the PWG for the method to be considered as a Type IV. Noting that the Procedural Manual did not preclude having a Type IV method when there was a Type I method identified for the same provision/commodity combination, this should be done on an exceptional basis only and should be fully justified.

38. CCMAS proceeded to consider this proposal.

39. Those delegations in favour of the proposal expressed the following views, some of which were a reiteration of view expressed at previous sessions of the Committee:

- the ISO | IDF method had limitations for use especially since the equipment and utensils were not widely available and costly and therefore not accessible to many countries;
- CCMAS should not only consider performance data but also look into applicability, availability and cost of methods in line with the criteria for selection of methods set out in the Procedural Manual;
- inclusion of the method as Type IV would allow countries to use the method if the ISO | IDF method was not available in line with one of the principles of Codex to ensure inclusiveness;
- the performance data which had been generated by inter-laboratory studies involving several accredited laboratories in South America, showed that the method is fit for purpose and meets the criteria to be selected as a method in CXS 234;
- the method could be endorsed as Type IV on the understanding that this was a very specific situation with regard to accessibility of the ISO | IDF method, and that a footnote could be introduced to explain this point. This would be similar to the approach taken in the fats and oils workable package review (see Agenda item 4).

40. Those delegations not in favour of the proposal at this time expressed the following views:

- there was already a method validated by an SDO available, i.e. ISO 5537 | IDF 26;
- the equipment was widely available and already in many countries worldwide and instructions on the construction of the equipment was available if cost of purchasing was a barrier;
- that if the method proposed in CX/MAS 23/42/3 Add.1 Appendix II, Annex 2 were endorsed, that more time was needed to review the performance data to confirm that the method was applicable to all the matrices identified and that the scope of the method would need to be reviewed. Some of the matrices were rich in lactose and the method might not be applicable for these matrices;
- that the method was similar to the IDF 26A:1993 method which had been withdrawn by the SDO;
- the method had been revoked on request of CCMMP because its reproducibility was determined not to be acceptable and replaced with the Type I methods. It could be listed exceptionally as Type IV when difficulties with equipment calibration are encountered;

[6] REP21/MAS, paras 36 - 41

- the method should be considered at CCMAS43 in order to consider the consequences/implications of including the method as Type IV;
- the importance of having good reproducibility for moisture determination since it affects other parameters such as protein determination.

41. The observer from IDF, reiterated statements made at CCMAS41 relating to the history of the method, the ready availability and limited cost of the equipment on the market and that there should not be an impediment to using the ISO | IDF method.

42. Noting that there was no hard rule to not endorse a Type IV method when there was a Type I, that the performance data had been submitted for review and indicated that the method was fit for purpose, CCMAS agreed to endorse the method as Type IV for the matrices identified in Appendix II Part 1.6. A footnote explaining that the method described in CXS 234 is listed as Type IV "*due to accessibility to equipment and calibration of the method ISO 5537 | IDF 26.*"

43. CCMAS agreed to consider the method for dairy permeate and whey powders at the next session and requested countries to submit further data to support this review. The scope of the method was also amended accordingly.

44. CCMAS agreed that the Codex Secretariat would issue a CL to request further information / data on the applicability of the method for dairy permeate and whey powders for consideration by the PWG on endorsement and CCMAS43.

Proposals by observer organisations

Determination of lactose and fat content in CXS 234 and update of ISO | IDF method for certain milk and milk products

45. CCMAS:

- endorsed ISO 22662 | IDF 198 as Type II for the determination of lactose in dairy permeate powders (Appendix II, Part 1); and
- the amendments as proposed in Appendix XI of CRD02 for milk and milk products.

Dietary Fibre Provisions in CXS 234

46. CCMAS noted that the proposal was withdrawn and that a proposal would be submitted to CCNFSDU for their consideration and possible referral to CCMAS.

OTHER MATTERS

47. CCMAS noted that a number of overarching issues arose during EWG discussions on workable packages (item 4) and in the PWG discussions and that the Committee should consider how to address these topics:

48. CCMAS took the following decisions on the overarching issues as follows:

A discussion and decision on the names and format used for the principles identified in CXS 234.

49. It was noted that the same principle is often identified in different ways, and there is not consistency in what information should be captured in the principle.

50. CCMAS agreed that Brazil would prepare a discussion paper to consider harmonization of names and format for principles identified in CXS 234 (an update of CX/MAS 17/38/6 Annex 3) as well as how to harmonize provision names (e.g. moisture content vs moisture).

51. An observer noted that when considering the names for the principles in CXS 234, consideration should be given to how much space there will be in the database and reminded that in terms of provisions, the provision names should be in line with the commodity standard.

The incorporation of nitrogen conversion factors in CXS 234.

52. CCMAS recalled an earlier decision that the Committee should not set conversion factors, which was the responsibility of the commodity committees, but noted that a consistent approach was needed on the placement of these conversions factors, either in CXS 234 or to remain solely in the commodity standard.

53. CCMAS agreed that Chile and Brazil would prepare a discussion paper to explore the best approach for the placement of nitrogen conversion factors.

Equivalency of Type I methods

54. CCMAS noted that CCMAS43 could consider whether a discussion paper should be developed to explore the matter.

Listing of Type IV methods in CXS 234 when a Type I method is listed for the same commodity and provision.

55. CCMAS recalled that at this session, an approach was taken to have both a Type I and Type IV method provided there was a justifiable and motivating reason. Using such an approach would require changes to the information document: *Comprehensive guidance for the process of submission, consideration and endorsement of methods for inclusion in CXS 234,* to describe this situation.

56. CCMAS agreed to establish an EWG chaired by Uruguay and co-chaired by Brazil, working in English to develop a discussion paper to:

- identify in CXS 234 all commodities and provisions where there are both Type I and Type IV methods listed;
- assess the reasons for both Type I and Type IV methods identified;
- discuss criteria and approaches for when Type I and Type IV methods can co-exist; and
- if necessary, make recommendations for changes to the information document and CXS 234.

Conclusion

57. CCMAS agreed to:

i. Submit the methods of analysis and sampling plans for adoption / revocation by CAC46 (Appendix II, Parts 1 and 2).

ii. Request CAC46 to revoke the *General Methods of Analysis for Contaminants* (CXS 228-2001).

iii. Inform relevant committees of the decisions taken at the session (paras 11, 12, 13, 15 – 17) and to refer the relevant requests to CCSCH (paras 23 and 25), CCAFRICA (para. 26), CCNASWP (para. 30), CCCF (paras 31 - 34) and CCASIA (paras 27 - 28).

iv. Update the Information Document: Sum of Components by the addition of the new example (para. 34); and inform CCCF of this decision.

v. Request the Codex Secretariat to issue CL(s) as identified in paras 11, 12, 34 and 44.

vi. Request Brazil and/or Chile to prepare discussion papers as identified in paras 50 and 53 for consideration by CCMAS43.

vii. Establish the following EWGs to prepare recommendations/discussion papers for consideration by CCMAS43:

a. An EWG chaired by the USA, working in English (see para. 13).

b. An EWG chaired by the USA and co-chaired by the UK, working in English (see para. 16).

c. an EWG chaired by Uruguay and co-chaired by Brazil (see para. 56).

viii. Re-establish the PWG on endorsement, chaired by the USA, the Netherlands, Hungary and Australia, subject to confirmation, working in English, to meet prior to the next session to consider all methods of analysis and sampling submitted by Codex Committees for endorsement, including the proposals on the workable packages: cereals, pulses and legumes, processed fruits and vegetables; fish and fishery products and fruit juices (see Agenda item 4); methods deferred by this session; and any other matters referred by other Codex Committees or submitted by members and observers.

REVIEW OF METHODS OF ANALYSIS IN CXS 234 (Agenda Item 4)

58. CCMAS recalled that the recommendations from the EWGs on the three workable packages: fats and oils, cereals, pulses and legumes; and processed fruits and vegetables had been considered by the PWG on endorsement. CCMAS considered the recommendations presented in CRD02.

FATS AND OILS WORKABLE PACKAGE (Agenda item 4.1)[7]

59. CCMAS:

- endorsed the methods as proposed (Appendix X, CRD02) including the footnote related to AOCS Cc 12-59 and its listing as a Type IV, even when a Type I exists for the same commodity and provision;
- noted that while revision work been undertaken by the EWG on methods related to provisions in the *Standard for Olive Oils and Olive Pomace Oils* (CXS 33-1981) (CRD08), review of these methods was suspended pending the ongoing revision of the *Standard for Olive Oils and Olive Pomace Oils*

[7] CL 2022/60/OCS-MAS; CX/MAS 23/42/4 (comments of Brazil, Canada, Chile, Colombia, Egypt, European Union, Iraq, Kenya, Norway, Peru, Philippines, Thailand, AOCS and ICUMSA)

(CXS 33-1981) in CCFO in accordance with the decision taken of CCMAS41; and

- noted that with these conclusions, the work on the review of methods for the fats and oils workable package had been concluded and thanked the Netherlands, chair of the EWG and members of the EWG for their work.

Conclusion

60. CCMAS agreed to submit the methods of analysis for adoption / revocation by CAC46 (Appendix II, Parts 1 and 2).

CEREALS, PULSES AND LEGUMES WORKABLE PACKAGE (Agenda item 4.2)[8]

61. CCMAS noted that the EWG on the cereals, pulses and legumes workable package had:

- completed work on the review of certain methods of analysis which were recommended for endorsement by the PWG (Appendix XI, Group 1 – CRD02);
- identified methods requiring additional follow up actions (Appendix XI, Group 2 – CRD02). Attention was drawn to the need for internationally validated suitable methods to replace the CAC/RM methods; and
- agreed to not taken action on new methods proposed by SDOs as updates and/or replacements for methods currently in CXS 234 (Appendix XI, Group 3 – CRD02) as consideration of new method proposals fell outside the purview of the EWG.

62. CCMAS further noted:

- that the methods proposed for aflatoxins in peanuts (raw / intended for further processing) should be revoked as there were already numeric performance criteria for methods adopted and published in the *General Standard for Contaminants and Toxins in Food and Feed* (CXS 193-1995). However, it was agreed that these performance criteria would need to be updated by CCCF and that this could be considered by the PWG on endorsement;
- confirmed that the methods for quinoa (a pseudo-cereal) should be further considered by the EWG taking into account validation data to be submitted for review.

Conclusion

63. CCMAS agreed:

- to submit the methods of analysis for adoption/revocation (Appendix II, Parts 1 and 2);
- to re-convene the EWG, chaired by Canada, working in English to continue review of the methods identified in Appendix II, Part 3; and
- agreed that new methods should be submitted in accordance with the *Comprehensive guidance for the process of submission, consideration and endorsement of methods for inclusion in CXS 234* for review by the PWG on endorsement. New methods proposals should be submitted in response to the CL to be issued by the Codex Secretariat.

PROCESSED FRUITS AND VEGETABLES WORKABLE PACKAGE (Agenda item 4.3)[9]

64. CCMAS noted:

- the methods for benzoic acid, sorbates, calcium and tin in processed fruits and vegetables would be retained in CXS 234, while numeric performance criteria are developed by the USA for consideration by the PWG on endorsement and CCMAS43;
- the work of the EWG had been completed and thanked the USA, chair of the EWG and the members of the EWG for their work; and
- the method for lead in certain processed fruits and vegetables should be removed from the table of methods for consideration by CCMAS (Appendix XI, CRD02) as numeric performance criteria had been agreed by the Committee (see para. 11) to replace the methods of analysis.

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8 CL 2023/13-MAS; CX/MAS 23/42/5; CX/MAS 23/42/5-Add.1(REV) (comments of Australia, Brazil, Egypt, European Union, Iraq, Jamaica, Paraguay, Philippines, Singapore and AACC International)

9 CL 2023/48 -MAS; CX/MAS 23/42/6; CX/MAS 23/42/6-Add.1 (comments of Australia, Brazil, Colombia, Egypt, European Union, Iraq, Paraguay and World Processing Tomato Council)

Conclusion

65. CCMAS: agreed to submit the methods of analysis for adoption and revocation by CAC46 (Appendix II, Parts 1 and 2).

Other matters

66. In view of the completion of the review of the fats and oils and processed fruits and vegetable workable packages, CCMAS agreed to:

    i. start the review of methods in the fish and fishery products; and fruit juices workable packages;

    ii. establish two EWGs

        a. chaired by Norway, and working in English to review the fish and fishery products workable package; and

        b. chaired by Germany, and working in English, to review the fruit juices workable package.

67. CCMAS recalled that the work of the review of methods in CXS 234 is to remove inconsistencies, make editorial corrections, check if the methods are still fit for purpose and to look at the Typing. To ease the work, new methods should not be considered in the EWG unless directly related to finding replacement methods for those no longer fit for purpose.

68. CCMAS reminded members and observers:

- they could present new methods for provisions in standards to active commodity committees for their consideration and submission to CCMAS as part of the endorsement process; or
- if the committee is adjourned sine die, that new methods could be submitted directly to CCMAS for review by the PWG on endorsement.

INFORMATION DOCUMENT: *GUIDELINES ON MEASUREMENT UNCERTAINTY* (CXG 54-2004) (Agenda Item 5)[10]

69. Germany, as Chair of the EWG, introduced the item and recalled that CCMAS39 (2018) had agreed to start new work on the revision of the *Guidelines on Measurement Uncertainty* (CXG 54-2004). In the process of the revision, CCMAS40 (2019), had agreed to keep the content of the Guidelines to the essentials for measurement uncertainty (MU) and that further information and explanations to facilitate their understanding and implementation would be provided in an information document. The first draft was presented to CCMAS41 (2021) which was subsequently revised by Germany based on the comments provided at that session to produce a new version as contained in Appendix I to CX/MAS 23/42/7. Key changes made were as follows:

- A few editorial changes were made throughout the document. A discussion of the Monte Carlo method (including an example) was added to Section 2.
- A new note regarding the case that precision depends on concentration was added at the end of Section 3.
- A paragraph regarding the necessity to perform a verification study was added at the end of Section 5.
- A new note regarding subsampling was added at the end of Section 7.
- The paragraph regarding confidence intervals for standard deviation estimates was rewritten in order to clarify the Excel syntax and the underlying mathematical expressions.
- A short paragraph summarizing the types of procedures described in the revised ISO 5725-3 and the new ISO TS 23471 was added at the beginning of Section 9.
- The references were updated.

70. The Delegation further explained that, following the request for comments through CL 2023/14-MAS, the document was further revised, based on comments submitted in reply to this CL, as presented in CRD04. Key changes made were as follows:

- Further editorial changes, including the addition of section headers for clearer articulation (e.g. Bottom-up approach: linear approximation, Bottom-up approach: Monte Carlo Method in Section 2).
- References (e.g. publication dates of ISO standards) were corrected.

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[10] CL 2023/14-MAS; CX/MAS 23/42/7; CX/MAS 23/42/7-Add.1 (Comments of Australia, Brazil, Canada, Chile, Egypt, European Union, Iraq, Japan, Mauritius, New Zealand, Paraguay, Philippines, Singapore and ICUMSA)

- A note regarding bias and CRMs was added in Section 3.
- A note regarding ISO 19036 was added in Section 7.
- A new subsection on contributions to uncertainty from subsampling and heterogeneity was added to Section 9.4. The section on fundamental variability is now a subsection to Section 9.4.
- A new section on procedures for identifying missing uncertainty contributions from MU estimates derived from precision data from collaborative studies was added (Section 9.5).

71. The Codex Secretariat clarified that information documents do not go through the Step Procedure, as opposed to Codex standards or guidelines, and as such, information documents were not adopted by the Codex Alimentarius Commission but remain available for internal use by the Committee or for public consultation on the Codex webpage following agreement by the Committee. She also noted that information documents could be considered as living documents subject to revisions when necessary. Therefore, further updates to this document could be made in future if required.

72. CCMAS noted general support for the information document as revised in CRD04.

73. Following a request to include a sentence in regard to the Monte Carlo Method (MCM) as an appropriate alternative approach to verify an already existing estimate of MU in certain circumstances, it was noted that the MCM could be an appropriate method as long as all the different components of MU are properly included in the equation/model. Following this explanation, it was agreed to include two additional sentences to indicate that (i) the MCM could be used to verify estimates of MU previously obtained via linear approximation but that (ii) if it cannot be ensured that all relevant sources of uncertainties are incorporated in the equation/model, then it is suggested to resort to the top-down approach.

Conclusion

74. CCMAS agreed:

i. with the revisions made to the information document as presented in CRD04 with the inclusion of the three sentences referred to in paragraph 73 above; and

ii. to publish the information document on the CCMAS webpage[11](Appendix III).

REVISION OF THE *GENERAL GUIDELINES ON SAMPLING* (CXG 50-2004) (Agenda Item 6)[12]

75. New Zealand, as Chair of the EWG, and also on behalf of the Co-Chair, Germany, introduced the item and summarized the work done on the revision of the Guidelines. The EWG/PWG Chair explained that the revised Guidelines reflect current scientific and statistical approaches for the development and evaluation of sampling plans.

76. The EWG Chair recalled that the Guidelines adopted in 2004 was considered very long and difficult to understand and implement and therefore of little use. In addition, sampling plans in commodity standards did not reflect CXG 50. In view of this, CCMAS39 (2018) had agreed to revise the Guidelines to provide a user-friendly approach intended to enable the development of sampling plans for provisions in Codex standards. She explained the revision included, amongst others, acceptance sampling plans for the inspection of isolated homogeneous lots in which the risks to consumers and producers are controlled, acceptance sampling plans for the control of the percentage nonconforming for homogeneous lots by attributes or by variables, for goods in bulk or individual items, some guidance on issues involved with the design of plans for bulk materials and other matters such as physical sampling, re-inspection and inhomogeneous lots. The appendices of the revised Guidelines contain a step-by-step guide for the selection of sampling plans and an explanation of the ISO sampling plans. She informed CCMAS that the Guidelines underwent several revisions since the approval of the new work by CAC41 (2018), through sessions of CCMAS, EWGs and webinars, and were adopted at Step 5 by CAC45 (2021). The EWG Chairs, New Zealand, and Germany, revised the Guidelines based on comments received in reply to CL 2023/15-MAS as contained in CRD03. She thanked Germany, Codex members and SDOs for their interest and assistance in the revision of the Guidelines.

77. The EWG Chair further recalled that, in addition to the revision of the Guidelines, a supporting information document namely e-book and sampling plan apps would be part of the next phase of the work to facilitate the understanding and implementation of the revised Guidelines. She explained that the information document would be completed during 2023-2024 and presented to CCMAS43 for finalization. The information document would address more detailed examples on measurement uncertainty (MU) and some practical examples of

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11 https://www.fao.org/fao-who-codexalimentarius/committees/committee/related-information-documents/en/?committee=CCMAS

12 CL 2023/15-MAS; CX/MAS 23/42/8; CX/MAS 23/42/8-Add.1 (Comments of Australia, Brazil, Ecuador, Egypt, European Union, Iraq, Japan, Mauritius, Philippines, Uganda)

sampling plans, amongst others.

78. In order to finalize the Guidelines, CCMAS agreed to establish an in-session WG under the chairmanship of New Zealand assisted by Germany to address some outstanding issues identified from the comments submitted in reply to CL 2023/15-MAS.

79. Following discussion in the in-session WG, CCMAS agreed to consider the Guidelines as revised in CRD25. CCMAS noted 4 key areas of revisions and other additional changes as summarized in CRD25. In addition, the Committee made the following decisions:

80. CCMAS:

- aligned the bulk material terminology for sampling plans with those in the sampling plans laid down in the *General Standard for Contaminants and Toxins in Food and Feed* (CXS 193-1995) and in doing so adjusted some terms to make it more general and inclusive consistent with the scope of the revised Guidelines e.g. remove the details around sample size as it varies depending on the produce being sampled, in addition to the reference to mill as it was specific to mycotoxins included "appropriate device" as a means of sample preparation in the definitions for test portion and laboratory sample, etc.;
- include a sentence in Appendix I to make it clear that examples are provided to assist in the design of sampling plans and not as definitive recommendations;
- corrected some discrepancies in the tables in Appendix II to the revised Guidelines; and
- noted that all references would be removed from the revised Guidelines consistent with the approach for Codex final texts.

Conclusion

81. CCMAS agreed to:

i. forward the revised *General Guidelines on Sampling* (CXG 50-2004) to CAC46 for adoption at Step 8 (Appendix IV);

ii. re-establish the EWG, chaired by New Zealand and co-chaired by Germany, working in English, to continue working on the information document namely the e-book with the sampling plans applications for consideration by CCMAS43; and

iii. inform relevant Codex committees of the revised *General Guidelines on Sampling* (CXG 50-2004) and request these committees to review their sampling plans in light of the revised Guidelines; and to remind Committees that sampling plans should be developed as needed in compliance with the *General Guidelines on Sampling* and not by a reference to CXG 50.

DISCUSSION PAPER ON CRITERIA TO SELECT TYPE II METHODS FROM MULTIPLE TYPE III METHODS (Agenda Item 7)[13]

82. The Observer from AOAC, speaking on behalf of Switzerland, recalled that CCMAS had agreed that rules for the selection of Type II methods from multiple Type III methods in CXS234 should be developed. He explained the process followed for development of the paper and recalled that CCMAS41 had agreed to circulate the proposed rules for selection of Type II methods from multiple Type III methods for comments and further revision by Switzerland and consideration by CCMAS42. The current paper had been revised taking into account comments received to CL 2022/23 -MAS.

83. He further explained that the proposed Rules had been tested for specific commodity-provision combinations with multiple Type III methods included in CXS234 and by the EWG on the review of fats and oils package. In addition, he reported that at the PWG on endorsement held prior to the session, it was agreed that Japan and AOAC would prepare additional criteria to ensure that methods that are selected are the ones that are the most feasible for use in most laboratories and that they do not require use of hazardous reagents under the section on *Prerequisites for inclusion in Codex standards for Type III chemical or physical methods*.

84. He proposed that CCMAS consider the proposal as follows:

"The method which is feasible in most of laboratories is preferred (e.g. use of more conventional instrument, etc.); and the methods that do not require hazardous reagents (e.g. heavy metals or organic chloride) is preferred."

[13] CX/MAS 23/42/9

Discussion

85. Diverse views were expressed on the additional criterion that the method which is feasible in most laboratories is preferred. While there was support for the proposal, there were also questions on the vagueness of the proposal, how to determine which methods were the most feasible and who would make such a determination.

86. CCMAS had extensive discussion on proposals to replace "feasibility" with "practicability and applicability", as used also in the Procedural Manual. However, questions remained on whether it was necessary to repeat criteria that were already covered by the *General criteria for the selection of methods of analysis* in the Procedural Manual. CCMAS was also reminded that the aim of the document was to provide guidance to select a Type II method from multiple Type III methods. It could be assumed that the multiple Type III methods had been selected in line with the *General criteria for the selection of methods of analysis* laid down in the Procedural Manual and as such they were considered fit for purpose, already adopted by CAC, and included in CXS234 therefore additional criteria would not be necessary.

87. Noting that the aim of the document was to provide guidance to select a Type II method from multiple Type III methods in CXS 234, CCMAS agreed that the section in question would be removed from the document as already covered by *General criteria for the selection of methods of analysis* in the Procedural Manual.

88. As a consequence, Table.1 was also amended to focus only on Type II methods and footnote 1 providing for flexibility in the application of the selection criteria was attached to the section on "Considerations for choosing a Type II method among multiple Type III methods".

89. CCMAS further took the following additional decisions in view of the discussion above:

- To change the title of the document to "guidance to select Type II methods from multiple Type III methods" since the document provided guidance that could be used to select Type II methods rather than a firm set of rules for such selection and to use this term where applicable throughout the document;
- to change the title of the section: "Decision rules for choosing a Type II method among multiple Type III methods" to "Considerations for choosing a Type II method among multiple Type III methods" as the points raised in this section were more considerations than fixed rules; and
- CCMAS also noted that these considerations were not in order of priority.

90. CCMAS noted a proposal to replace two criteria related to selectivity and best precision data (if the precision difference is relevant to the question asked) with a criterion referring to the practicability and applicability of the method under normal laboratory conditions. It was further noted that the qualifier "relevant" was subjective and should be replaced by "significant"; that "accuracy" was more appropriate than "precision" and that "recovery" should also be added as a criterion to select a Type II method from Type III methods.

91. Members not in agreement with these proposals indicated the following:

- the measurable performance parameters cannot be replaced by the practicability and applicability of the method although they were also important criteria to consider when selecting a Type II method from Type III methods due to the different capabilities of countries and regions;
- it would not be appropriate to consider "practicability and applicability" at the stage when a Type II method is selected as it is already considered when a method becomes Type III;
- recovery was already considered in the selection of Type III methods and, for the selection of a Type II method from Type III methods, precision was more important than accuracy as, in choosing between two methods which would be the dispute method, the precision of these methods would be more critical in order to obtain consistent results;
- accuracy was already considered in the criterion where a certified reference material (CRM) is used.

92. An Observer noted that "relevant" would be a more reasonable assumption than "significant" as it would be difficult to define a significant difference which envisage statistics and equivalence considerations which imply a lot of work that might not be possible to carry out in CCMAS.

93. A member further noted that most of the parameters mentioned are already considered in the Procedural Manual for the selection of methods, however the discussion was not about selecting a reference method that is applicable for use but about selecting a Type II reference method among multiple Type III methods to resolve a trade dispute and therefore it was important to have the best precision to give that discrimination, the various matrices, the selectivity and the CRM used to support its accuracy.

94. Based on the above considerations, CCMAS agreed to retain the criteria on selectivity and precision and to retain the term "relevant" as more practical; and to include an additional consideration that the method should be practicable and applicable under normal laboratory conditions.

Conclusion

95. CCMAS agreed to the Guidance on the Selection of Type II methods from multiple Type III methods as amended (Appendix V) for inclusion in the CCMAS information document: *Comprehensive guidance for the process of submission, consideration and endorsement of methods for inclusion in CXS 234*.

REPORT OF AN INTER-AGENCY MEETING ON METHODS OF ANALYSIS (Agenda Item 8)

96. The Observer of the United States Pharmacopeial Convention (USP), as Chair of the Inter-Agency Meeting (IAM), introduced the report of the IAM and highlighted the various issues discussed in the IAM with respect to the work of CCMAS and other related matters i.e. update on CXS 234 method review progress, other CCMAS related issues, such as selection of Type II method from multiple Type III methods, as well as updates on events and ongoing work carried out by the SDOs as described in CRD24.

97. CCMAS noted that several of the issues raised in CRD24, had been considered under relevant agenda items.

Conclusion

98. CCMAS thanked the members of IAM for their valuable contribution to the work of the Committee.

OTHER BUSINESS AND FUTURE WORK (Agenda Item 9)

99. CCMAS noted that there was no other business for discussion.

100. The Codex Secretariat noted that it might be useful for the Committee to start a discussion on the future of the Committee and to explore new areas of work that might need the attention of CCMAS especially with the completion of the work on the *General Guidelines on Sampling* (CXG 50-2004) and the *Guidelines on Measurement Uncertainty* (CXG 54-2004). She invited delegates to think about issues that could be brought to CCMAS43 for discussion and to consider reviewing other CCMAS texts with the view of determining whether they were still fit for purpose, needed revision, or should be revoked.

Conclusion

101. CCMAS noted the proposal of the Codex Secretariat.

DATE AND PLACE OF NEXT SESSION (Agenda Item 10)

102. CCMAS was informed that its 43rd Session was tentatively scheduled to take place on 13 – 17 May 2024 in Budapest. The final arrangements being subject to confirmation by the Host Country and the Codex Secretariat.

APPENDIX I

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LISTE DES PARTICIPANTS
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APPENDIX II*

Part 1. METHODS OF ANALYSIS AND SAMPLING PLANS FOR ADOPTION BY CAC46

1.1. CODEX COMMITTEE ON NUTRITION AND FOODS FOR SPECIAL DIETARY USES (CCNFSDU)

1.2. FAO/WHO CCORDINATING COMMITTEE FOR AFRICA (CCAFRICA24)

1.3. FAO/WHO COORDINATING COMMITTEE FOR ASIA (CCASIA)

1.4. FAO/WHO COORDINATING COMMITTEE FOR NORTH AMERICA AND THE SOUTHWEST PACIFIC (CCNASWP)

1.5. CODEX COMMITTEE ON CONTAMINANTS IN FOODS (CCCF)

1.6. MILK AND MILK PRODUCTS

1.7. FATS AND OILS

1.8. CEREALS, PULSES AND LEGUMES

1.9. PROCESSED FRUITS AND VEGETABLES

Part 2. METHODS OF ANALYSIS FOR REVOCATION BY CAC46

2.1 CODEX COMMITTEE ON CONTAMINANTS IN FOODS (CCCF)

2.2 FATS AND OILS

2.3 CEREALS, PULSES AND LEGUMES

2.4 PROCESSED FRUITS AND VEGETABLES

2.5 MILK AND MILK PRODUCTS

Part 3. CEREALS, PULSES AND LEGUMES WORKABLE PACKAGE FOR CONSIDERATION BY THE ELECTRONIC WORKING GROUP ON CEREALS

* *Corrections have been made to this Appendix by removing duplications; addition of footnotes to the methods for titre in named animal fat and for unsaponifiable matter in named vegetables oils and additional methods for revocation from specific commodity standards which had been inadvertently omitted*.

PART 1

METHODS OF ANALYSIS AND SAMPLING FOR ADOPTION BY CAC46

(Methods and performance criteria are for inclusion in CXS 234-1999: changes indicated in **bold** or underlined font).

1.1 CODEX COMMITTEE ON NUTRITION AND FOODS FOR SPECIAL DIETARY USES (CCNFSDU)

Methods of analysis for provisions in the Standard for Infant Formula and Formulas for Special Medical Purposes Intended for Infants (CXS 72-1981)

Commodity	Provision	Method	Principle	Type
Infant Formula	**Vitamin B12**	**AOAC 2014.02**	**LC-UV**	**III**
	Total amino acids (excluding taurine and tryptophan) **For use according to Section 3.1.3 (a) footnotes 3 and 4 of CXS 72-1981**	**AOAC 2018.06 / ISO 4214 | IDF 254 / AACC 07-50.01**	**UHPLC-UV**	**II**
	Tryptophan **For use according to Section 3.1.3 (a) footnotes 3 and 4 of CXS 72-1981**	**AOAC 2017.03**	**HPLC**	**II**

1.2 FAO/WHO COORDINATING COMMITTEE FOR AFRICA (CCAFRICA24)

Methods of analysis and sampling for provisions in the Regional Standard for Dried Meat (CXS 350-2022)

Provision	Method	Principles	Type
Moisture Content	AOAC 950.46B	Gravimetry	I
Total Fat	ISO 1443	Gravimetry	I
~~Nitrogen~~ **Protein*** ***nitrogen-to-protein conversion factor = 6.25**	ISO 937*	**Calculation and** Titrimetry	I
Chloride as Sodium Chloride (≥ 1.0%)	ISO 1841-1	**Titrimetry** (Volhard method)	III
Chloride as Sodium Chloride (≥ 0.25%)	ISO 1841-2	**Titrimetry** (Potentiometry)	II
Ash	ISO 936	Gravimetry	I
Water Activity	ISO 18787	Electrometry	II

1.3 FAO/WHO COORDINATING COMMITTEE FOR ASIA (CCASIA)

Methods of analysis and sampling for provisions in the draft Regional Standard for Soybean Products Fermented with Bacillus Species

Commodity	Provision	Method	Principles	Type
Natto	Moisture content	AOAC 925.09	Gravimetry	I
Natto	Protein content (*nitrogen-to-protein conversion factor = 5.71)	AOAC 988.05	Titrimetry, (Kjeldahl)	I
Natto	Lipid Content 4 g quantity of samples	AOAC 963.15	Gravimetry (Soxhlet)	I
Cheonggukjang	Moisture content	AOAC 934.01	Gravimetry	I
Cheonggukjang	Protein content (*nitrogen-to-protein conversion factor = 5.71)	AOAC 988.05	Titrimetry, (Kjeldahl)	I
Cheonggukjang	Lipid Content 5 g quantity of samples	AOAC 963.15	Gravimetry (Soxhlet)	I
Thua Nao	Moisture content	AOAC 925.09	Gravimetry	I
Thua Nao	Protein content (*nitrogen-to-protein conversion factor = 5.71)	AOAC 988.05	Titrimetry, (Kjeldahl)	I

ANNEX A: Methods of analysis for provisions in the draft Regional Standard for Cooked Rice Wrapped in Plant Leaves

Determination of Peroxide Value

Extraction of Oils from the Product

Apparatus

(a) Rotary evaporator

(b) Water bath

Extraction

Remove the product package and plant leaves, etc. take out the edible part of the representative sample, crush it and put it in a homogenizer or glass mortar, and grind it continuously to make the sample fully mashed and mixed well, and then put it in the wide-mouth bottle, and add 2 to 3 times the sample volume of petroleum ether (boiling range: 30°C-60°C). After fully mixing, stopper the bottle and leave for more than 12 hours. Filter all the solution with a funnel filled with anhydrous sodium sulphate into a round-bottom flask. Rinse the residue in the wide-mouth bottle with petroleum ether. Repeat the filtration once with a new anhydrous sodium sulphate funnel, if the filtrate is not clear enough. Evaporate the petroleum ether in the round-bottom flask under reduced pressure on a rotary evaporator at below 40°C, and the residue is the test sample. A sufficient number of representative samples should be selected to ensure that not less than 8 grams of the test sample can be obtained. The test sample should be tested as soon as possible.

Determination

Commodity	Provision	Method	Principles	Type
Cooked rice wrapped in plant leaves	**Peroxide Value**	**ISO 3960 / AOCS Cd 8b-90**	**Titrimetry**	**IV**

1.4 FAO/WHO COORDINATING COMMITTEE FOR NORTH AMERICA AND THE SOUTHWEST PACIFIC (CCNASWP)

Methods of analysis and sampling for provisions in the draft Regional Standard for Fermented Noni Fruit Juice

Provision	Method	Principle	Type
Brix value (Soluble solids)	**AOAC 983.17 / EN 12143 / IFUMA 8 / ISO 2173**	**Refractometry**	**IV**
Identification of scopoletin	**Annex B***	**Solid phase extraction and thin layer chromatography**	**IV**
Identification of deacetylasperulosidic acid	**Annex C***	**Thin layer chromatography**	**IV**

ANNEX B. IDENTIFICATION OF SCOPOLETIN

1. PREPARATION OF SAMPLES

Noni fruit juice is filtered through a 0.45 µm membrane filter and then purified by solid-phase extraction (SPE) with Waters OASIS® HLB 6cc 200 mg extraction cartridges (or similar solid-phase extraction cartridge), after first equilibrating with methanol (5 mL) followed by deionized water (5 mL). The filtered juice samples (3 mL) are then loaded onto the equilibrated cartridge and washed with 5% methanol (MeOH) in deionized water (5 mL). The cartridges are allowed to dry under flow of air for 5 mins and then, eluted with MeOH (3mL). The MeOH eluate is retained for TLC analysis. The SPE flow rates of equilibration, wash and elution solvents through the cartridge is approximately 1 drop per second.

2. PREPARATION OF REFERENCE STANDARD

2.1 A reference standard is prepared by dissolving 0.1 mg Scopoletin in 1 millilitre of methanol.

2.2 Alternately, certified *Morinda citrifolia* reference plant material may be prepared in the same manner as the samples to be analyzed. The certified *Morinda citrifolia* reference material should be from the same part of the plant as the samples to be analyzed.

3. IDENTIFICATION

3.1 THIN LAYER CHROMATOGRAPHY

Spot 5 microliters of sample solutions and reference standard solution on a silica gel 60 F254 thin layer chromatography (TLC) plate. After spotting the plates are dried at 110°C for 15 minutes in a drying oven. Develop the plate with a mobile phase of dichloromethane:methanol (19:1, v/v). View bright fluorescent blue colours on developed plate under UV lamp, 365 nm. Identify Scopoletin in samples by comparing Rf values and colours to the standard.

Annex C. IDENTIFICATION OF DEACETYLASPERULOSIDIC ACID

1. PREPARATION OF SAMPLES

Noni fruit juice is filtered through a 0.45 µm membrane filter and diluted 1:1 with MeOH.

2. PREPARATION OF REFERENCE STANDARD

2.1 A reference standard is prepared by dissolving 1 mg deacetylasperulosidic acid in 1 millilitre of methanol.

2.2 Alternately, certified *Morinda citrifolia* reference plant material may be prepared in the same manner as the samples to be analysed. The certified *Morinda citrifolia* reference material should be from the same part of the plant as the samples to be analysed.

3. PREPARATION OF p-ANISALDEHYDE SOLUTION

Anisaldehyde solution was prepared by dissolving 2g of p-anisaldehyde in 96 mL of ethanol with stirring. The solution was then acidified through dropwise addition of concentrated sulfuric acid (4 mL).

4. IDENTIFICATION

4.1 THIN LAYER CHROMATOGRAPHY

Spot 5 microliters of sample solutions and reference standard solution on a silica gel 60 F254 thin layer chromatography (TLC) plate, previously dried at 110°C for 15 minutes in a drying oven. After spotting samples are again dried at 110ºC or through application of heat via a heat gun for a period of 8-10 seconds. The TLC plates are developed with a mobile phase of dichloromethane: methanol: water (13:6:1, v/v/v). Upon completion of elution, the plate is air dried and developed by spraying with 2% anisaldehyde / 4% sulfuric acid in ethanol (EtOH) solution and then heat in oven at 110°C for 1-5 minutes to reveal and maximise the blue colour. Identify deacetylasperulosidic in samples by comparing spot Rf values and colour with reference standard solution on same TLC plate.

1.5 CODEX COMMITTEE ON CONTAMINANTS IN FOODS (CCCF)

Table I: Numeric performance criteria for lead and cadmium for endorsement and inclusion in the *Recommended Methods of Analysis and Sampling* (CXS 234-1999)

Numeric performance criteria for lead and cadmium in foods

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria[1]	Principle
Natural mineral waters	lead	0.01	0.006 - 0.014	0.002	0.004	44	60-115%		
Infant formula, formula for special medical purposes intended for infants and follow-up formula	lead	0.01	0.006 - 0.014	0.002	0.004	44	60-115%		
Milk	lead	0.02	0.011 - 0.029	0.004	0.008	44	60-115%		
Secondary milk products	lead	0.02	0.011 - 0.029	0.004	0.008	44	60-115%		
Fruit juices, except juices exclusively from berries and other small fruits	lead	0.03	0.017 - 0.043	0.006	0.012	44	60-115%		
Fat spreads and blended spreads	lead	0.04	0.022 - 0.058	0.008	0.016	44	60-115%		
Grape juice	lead	0.04	0.022 - 0.058	0.008	0.016	44	60-115%		
Canned chestnuts and canned chestnuts puree	lead	0.05	0.028 - 0.072	0.010	0.020	44	60-115%		

[1] Example methods will be reviewed by CCMAS43

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria[1]	Principle
Fruit juices obtained exclusively from berries and other small fruits, except grape juice	lead	0.05	0.028 - 0.072	0.010	0.020	44	60-115%		
Fruiting vegetables, except fungi and mushrooms	lead	0.05	0.028 - 0.072	0.010	0.020	44	60-115%		
Preserved tomatoes	lead	0.05	0.028 - 0.072	0.010	0.020	44	60-115%		
Edible fats and oils	lead	0.08	0.045 - 0.115	0.016	0.032	44	60-115%		
Berries and other small fruits, except cranberry, currant, and elderberry	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Brassica vegetables, except kale and leafy Brassica vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Bulb vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Canned fruits	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Canned vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Fruits, except cranberry, currants, and elderberry	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria[1]	Principle
Legume vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Meat and fat of poultry	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Meat of cattle, pigs and sheep	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Pickled cucumbers (cucumber pickles)	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Poultry, edible offal of	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Pulses	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Root and tuber vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Wine from grapes harvested after July 2019	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Fortified / Liqueur wine from grapes harvested after 2019	lead	0.15	0.05 - 0.25	0.015	0.03	43	80-110%		
Pig, edible offal of	lead	0.15	0.05 - 0.25	0.015	0.03	43	80-110%		
Cattle, edible offal of	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%		

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria[1]	Principle
Cereal grains, except buckwheat, cañihua and quinoa	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%		
Cranberry	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%		
Currants	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%		
Elderberry	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%		
Wine (wine and fortified / liqueur wine) made from grapes harvested before July 2019	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%		
Fish	lead	0.3	0.13 - 0.47	0.03	0.06	38	80-110%		
Fresh farmed mushrooms (common mushrooms (*Agaricus bisporous*), shiitake mushrooms (*Lentinula edodes*), and oyster mushrooms (*Pleurotus ostreatus*))	lead	0.3	0.13 - 0.47	0.03	0.06	38	80-110%		
Leafy vegetables, except spinach	lead	0.3	0.13 - 0.47	0.03	0.06	38	80-110%		
Jams, jellies, and marmalades	lead	0.4	0.18 - 0.62	0.04	0.08	37	80-110%		

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria[1]	Principle
Mango chutney	lead	0.4	0.18 - 0.62	0.04	0.08	37	80-110%		
Table olives	lead	0.4	0.18 - 0.62	0.04	0.08	37	80-110%		
Salt, food grade	lead	1	0.5 - 1.5	0.1	0.2	32	80-110%		
Natural mineral waters	cadmium	0.003	0.0017 - 0.0043	0.0006	0.0012	44	40-120%		
Brassica vegetables, except Brassica leafy vegetables	cadmium	0.05	0.03 - 0.07	0.01	0.02	44	60-115%		
Bulb vegetables	cadmium	0.05	0.03 - 0.07	0.01	0.02	44	60-115%		
Fruiting vegetables, except tomatoes and edible fungi	cadmium	0.05	0.03 - 0.07	0.01	0.02	44	60-115%		
Cereal grains, except buckwheat, cañihua, quinoa, wheat and rice	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Legume vegetables	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Pulses, except soya bean (dry)	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Root and tuber vegetables, except celeriac	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria[1]	Principle
Stalk and stem vegetables	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Leafy vegetables	cadmium	0.2	0.08 - 0.32	0.02	0.04	41	80-110%		
Wheat (common wheat, durum wheat, spelt and emmer)	cadmium	0.2	0.08 - 0.32	0.02	0.04	41	80-110%		
Chocolate containing or declaring < 30% total cocoa solids on a dry matter basis	cadmium	0.3	0.13 - 0.47	0.03	0.06	38	80-110%		
Rice, polished	cadmium	0.4	0.18 - 0.62	0.04	0.08	37	80-110%		
Salt, food grade	cadmium	0.5	0.23 - 0.77	0.05	0.10	36	80-110%		
Chocolate containing or declaring ≥30% to <50% total cocoa solids on a dry matter basis	cadmium	0.7	0.35 - 1.05	0.07	0.14	34	80-110%		

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria[1]	Principle
Chocolate containing or declaring ≥50% to <70% total cocoa solids on a dry matter basis, including sweet chocolate, Gianduja chocolate, semi – bitter table chocolate, Vermicelli chocolate / chocolate flakes, and bitter table chocolate	cadmium	0.8	0.40 - 1.20	0.08	0.16	33	80-110%		
Chocolate containing or declaring ≥70% total cocoa solids on a dry matter basis, including sweet chocolate, Gianduja chocolate, semi – bitter table chocolate, Vermicelli chocolate / chocolate flakes, and bitter table	cadmium	0.9	0.46 - 1.34	0.09	0.18	33	80-110%		
Cephalopods	cadmium	2	1.1 - 2.9	0.2	0.4	29	80-110%		
Marine bivalve molluscs (clams, cockles and mussels), except oysters and scallops	cadmium	2	1.1 - 2.9	0.2	0.4	29	80-110%		

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

(For inclusion in CXS 193-1995)

Sampling plans and performance criteria for aflatoxin (AFB1+AFB2+AFG1+AFG2) in maize grain, destined for further processing.

Maximum level	15 µg/kg AFB1+AFB2+AFG1+AFG2
Increments	Increments of 100g, 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	≥5 kg
Number of laboratory samples	1
Test portion	25 g
Method	Selected according to the established performance criteria in Table 3
Decision rule	If the sum of test results of AFB1, AFB2, AFG1 and AFG2 for the laboratory sample is equal to or less than 15 µg/kg, accept the lot. Otherwise, reject the lot.

Sampling plans and performance criteria for aflatoxin (AFB1+AFB2+AFG1+AFG2) in flour meal, semolina and flakes derived from maize

Maximum level	10 µg/kg AFB1+AFB2+AFG1+AFG2
Increments	10 x 100g
Sample preparation	dry grind with a suitable mill (particles smaller than 0.85 mm – 20 mesh), if necessary for coarse samples
Laboratory sample weight	1 kg
Number of laboratory samples	1
Test portion	25g
Method	Selected according to the established performance criteria in Table 3
Decision rule	If the sum of test results of AFB1, AFB2, AFG1 and AFG2 for the laboratory sample is equal to or less than 10 µg/kg, accept the lot. Otherwise, reject the lot

Sampling plans and performance criteria for aflatoxin (AFB1+AFB2+AFG1+AFG2) in husked rice

Maximum level	20 µg/kg AFB1+AFB2+AFG1+AFG2
Increments	Increments of 100g, 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	≥5 kg
Number of laboratory samples	1
Test portion	25g
Method	Selected according to the established performance criteria in Table 3
Decision rule	If the sum of test results of AFB1, AFB2, AFG1 and AFG2 for the laboratory sample is equal to or less than 20 µg/kg, accept the lot. Otherwise, reject the lot

Sampling plans and performance criteria for aflatoxin (AFB1+AFB2+AFG1+AFG2) in polished rice

Maximum level	5 µg/Kg AFB1+AFB2+AFG1+AFG2
Increments	Increments of 100g, 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	≥5 kg
Number of laboratory samples	1
Test portion	25g
Method	Selected according to the established performance criteria in Table 3
Decision rule	If the sum of test results of AFB1, AFB2, AFG1 and AFG2 for the laboratory sample is equal to or less than 5 µg/kg, accept the lot. Otherwise, reject the lot

Sampling plans and performance criteria for aflatoxin (AFB1+AFB2+AFG1+AFG2) in sorghum

Maximum level	10 µg/kg AFB1+AFB2+AFG1+AFG2
Increments	Increments of 100g, 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 size	≥5 kg
Number of laboratory weight	1
Test portion	25g
Method	Selected according to the established performance criteria in Table 3
Decision rule	If the sum of test results of AFB1, AFB2, AFG1 and AFG2 for the laboratory sample is equal to or less than 10 µg/kg, accept the lot. Otherwise, reject the lot

Sampling plans and performance criteria for aflatoxin (AFB1+AFB2+AFG1+AFG2) in cereal-based food for infants and young children

Maximum level	5 µg/kg AFB1+AFB2+AFG1+AFG2
Increments	10 x 100g
Sample preparation	dry grind with a suitable mill (particles smaller than 0.85 mm – 20 mesh), if necessary for coarse samples
Laboratory sample weight	1 kg
Number of laboratory samples	1
Test portion	25g
Method	Selected according to the established performance criteria in Table 3
Decision rule	If the sum of test results of AFB1, AFB2, AFG1 and AFG2 for the laboratory sample is equal to or less than 5 µg/kg, accept the lot. Otherwise, reject the lot

Sampling plans and performance criteria for aflatoxin (AFB1+AFB2+AFG1+AFG2) in cereal-based food for infants and young children destined for food aid programs

Maximum level	10 µg/kg AFB1+AFB2+AFG1+AFG2
Increments	10 x 100g
Sample preparation	dry grind with a suitable mill (particles smaller than 0.85 mm – 20 mesh), if necessary for coarse samples
Laboratory sample size	1 kg
Number of laboratory weight	1
Test portion	25g
Method	Selected according to the established performance criteria in Table 3
Decision rule	If the sum of test results of AFB1, AFB2, AFG1 and AFG2 for the laboratory sample is equal to or less than 10 µg/kg, accept the lot. Otherwise, reject the lot

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	It is defined by an aflatoxin test procedure and an accept/reject level. An aflatoxin test procedure consists of three steps: sample selection, sample preparation and analysis or aflatoxin 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 grains, shelled cereal grains and cereal-based products 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 aflatoxin for chemical analysis.

SAMPLING PLAN DESIGN CONSIDERATIONS

MATERIAL TO BE SAMPLED

1. Each lot of cereal grains and cereal-based products, which is to be examined for AFs, 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 cereal grains sublots according to lot weight – Maize grain, sorghum, polished rice and husked rice

Lot weight (t)	Maximum weight or minimum number of sublots	Number of incremental samples	Minimum laboratory sample weight (kg)
≥ 1500	500 tons	100	5
> 300 and < 1500	3 sublots	100	5
≥ 100 and ≤ 300	100 tons	100	5
≥ 50 and < 100	2 sublots	100	5
< 50	-	3-100*	5

*see Table 2

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 size by a maximum of 20%.

INCREMENTAL SAMPLE

3. The suggested minimum size of the incremental sample of cereal grains and cereal-based products should be 100 g for lots ≥ 0.5 tons.

4. For lots less than 50 tons of cereal grains and cereal-based products, 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 5 kg. Table 2 may be used to determine the number of incremental samples to be taken.

Table 2. Number of incremental samples of cereal grains to be taken depending on the weight of the lot- Maize grain, sorghum, polished rice and husked rice

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

STATIC LOTS

5. A static lot can be defined as a large mass cereal grains and cereal-based products 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 grains and cereal-based products 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.

6. 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.

7. 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 size (LT), incremental sample size (IS), aggregate sample size (AS) and the individual packing size (IP), as follows:

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

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

DYNAMIC LOTS

9. Representative aggregate samples can be more easily produced when selecting incremental samples from a moving stream of cereal grains and cereal-based products 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).

10. 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.

11. 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.

12. The size of the aggregate sample (S) in kg, taken from a lot by a crosscut 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).

13. 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

14. 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

15. 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

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

HOMOGENIZATION - GRINDING

17. As the distribution of aflatoxin 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.

18. 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 is minimized. After grinding, the grinder should be cleaned to prevent aflatoxin cross-contamination.

TEST PORTION

19. The suggested weight of the test portion taken from the comminuted laboratory sample should be approximately 25 g. If the laboratory sample is prepared using a liquid slurry, the slurry should contain 25 g.

20. 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.

ANALYTICAL METHODS

21. 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 is shown in Table 3. Utilizing this approach, laboratories would be free to use the analytical method most appropriate for their facilities.

Table 3. Method criteria for total aflatoxins in cereals, considering AFB1: AFB2:AFG1:AFG2 of 1:1:1:1.

Commodity	Analyte	ML (µg/kg)	LOD (µg/kg)	LOQ (µg/kg)	Precision (%)	Minimal applicable range (µg/kg)	Recovery (%)
Maize grain	AF B1+B2+G1+G2	15	≤ 3	≤ 6	<44	8.4 - 21.6	60-115
	AFB1	-	≤0.75	≤ 1.5	<44	2.1 – 5.4	40-120
	AFB2	-	≤0.75	≤1.5	<44	2.1 – 5.4	40-120
	AFG1	-	≤0.75	≤ 1.5	<44	2.1 – 5.4	40-120
	AFG2	-	≤0.75	≤ 1.5	<44	2.1 – 5.4	40-120
Maize flour, meal, semolina and flakes derived from maize; Sorghum grain; cereal-based foods for infants and young children for food aid programs	AF B1+B2+G1+G2	10	≤2	≤4	<44	5.6 - 14.4	60-115
	AFB1	-	≤0.5	≤1.0	<44	1.4 - 3.6	40-120
	AFB2	-	≤0.5	≤1.0	<44	1.4 - 3.6	40-120
	AFG1	-	≤0.5	≤1.0	<44	1.4 - 3.6	40-120
	AFG2	-	≤0.5	≤1.0	<44	1.4 - 3.6	40-120
Husked Rice	AF B1+B2+G1+G2	20	≤4	≤8	<44	11.2 - 28.8	60-115
	AFB1	-	≤1.0	≤2.0	<44	2.8 – 7.2	40-120
	AFB2	-	≤1.0	≤2.0	<44	2.8 – 7.2	40-120
	AFG1	-	≤1.0	≤2.0	<44	2.8 – 7.2	40-120
	AFG2	-	≤1.0	≤2.0	<44	2.8 – 7.2	40-120
Polished Rice; Cereal-based food for infants and young children	AF B1+B2+G1+G2	5	≤1	≤2	<44	2.8 - 7.2	40-120
	AFB1	-	≤0.25	≤0.5	<44	0.7 – 1.8	40-120
	AFB2	-	≤0.25	≤0.5	<44	0.7 – 1.8	40-120
	AFG1	-	≤0.25	≤0.5	<44	0.7 – 1.8	40-120
	AFG2	-	≤0.25	≤0.5	<44	0.7 – 1.8	40-120

1.6 MILK AND MILK PRODUCTS

(* footnotes 13 and 14 to the methods for milk solids-not-fat and water (moisture) are the equivalent footnotes as they appear in CXS 234-1999)

Commodity	Provision	Method	Principle	Type
Dairy permeate powders	**Lactose**	**ISO 22662 | IDF 198**	**High performance liquid chromatography**	**II**
Blend of evaporated skimmed milk and vegetable fat	Total fat	**ISO 23318 | IDF 249**	Gravimetry (Röse-Gottlieb)	I
Blend of evaporated skimmed milk and vegetable fat	Milk solids-not-fat[13] (MSNF)	ISO 6731 | IDF 21 and **ISO 23318 | IDF 249**	Calculation from total solids content and fat content Gravimetry, drying at 102°C and Gravimetry (Röse-Gottlieb)	I
Blend of evaporated skimmed milk and vegetable fat	Milk protein in MSNF[13]	ISO 6731 | IDF 21 and **ISO 23318 | IDF 249** and ISO 8968-1 | IDF 20-1	Calculation from total solids content, fat content and protein content Gravimetry, drying at 102°C and Gravimetry (Röse-Gottlieb) and Titrimetry (Kjeldahl)	IV
Blend of evaporated skimmed milk and vegetable fat	Milk protein in MSNF[13]	ISO 6731 | IDF 21 and **ISO 23318 | IDF 249** and AOAC 991.20	Calculation from total solids content, fat content and protein content Gravimetry, drying at 102°C and Gravimetry (Röse-Gottlieb) and Titrimetry (Kjeldahl)	IV
Reduced fat blend of evaporated skimmed milk and vegetable fat	Total fat	**ISO 23318 | IDF 249**	Gravimetry (Röse-Gottlieb)	I

Commodity	Provision	Method	Principle	Type
Reduced fat blend of evaporated skimmed milk and vegetable fat	Milk solids-not-fat (MSNF) [13]	ISO 6731 | IDF 21 and **ISO 23318 | IDF 249**	Calculation from total solids content and fat content Gravimetry, drying at 102°C and Gravimetry (Röse-Gottlieb)	I
Reduced fat blend of evaporated skimmed milk and vegetable fat	Milk protein in MSNF[13]	ISO 6731 | IDF 21 and **ISO 23318 | IDF 249** and ISO 8968-1 | IDF 20-1	Calculation from total solids content, fat content and protein content Gravimetry, drying at 102°C and Gravimetry (Röse-Gottlieb) and Titrimetry (Kjeldahl)	IV
Reduced fat blend of evaporated skimmed milk and vegetable fat	Milk protein in MSNF[13]	ISO 6731 | IDF 21 and **ISO 23318 | IDF 249** and AOAC 991.20	Calculation from total solids content, fat content and protein content Gravimetry, drying at 102°C and Gravimetry (Röse-Gottlieb) and Titrimetry (Kjeldahl)	IV
Blend of skimmed milk and vegetable fat in powdered form	Total fat	**ISO 23318 | IDF 249**	Gravimetry (Röse-Gottlieb)	I
Blend of skimmed milk and vegetable fat in powdered form	**Water[14] (moisture)**	**Described in Annex D***	**Gravimetry, drying at 102°C**	**IV**
Blend of skimmed milk and vegetable fat in powdered form	Milk protein in MSNF[13]	ISO 5537 | IDF 26 and **ISO 23318 | IDF 249** and ISO 8968-1 | IDF 20-1	Calculation from total solids content, fat content and protein content Gravimetry, drying at 87°C and Gravimetry (Röse-Gottlieb) and Titrimetry (Kjeldahl)	IV

Commodity	Provision	Method	Principle	Type
Blend of skimmed milk and vegetable fat in powdered form	Milk protein in MSNF[13]	**Described in Annex D* and ISO 23318 | IDF 249 and ISO 8968-1 | IDF 20-1**	**Calculation from total solids content, fat content and protein content Gravimetry, drying at 102°C and Gravimetry (Röse-Gottlieb) and Titrimetry (Kjeldahl)**	**IV**
Blend of skimmed milk and vegetable fat in powdered form	Milk protein in MSNF[13]	ISO 5537 | IDF 26 and **ISO 23318 | IDF 249** and AOAC 991.20	Calculation from total solids content, fat content and protein content Gravimetry, drying at 87°C and Gravimetry (Röse-Gottlieb) and Titrimetry (Kjeldahl)	IV
Blend of skimmed milk and vegetable fat in powdered form	**Milk protein in MSNF[13]**	**Described in Annex D* and ISO 23318 | IDF 249 and AOAC 991.20**	**Calculation from total solids content, fat content and protein content Gravimetry, drying at 102°C and Gravimetry (Röse-Gottlieb) and Titrimetry (Kjeldahl)**	**IV**
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Total fat	**ISO 23318 | IDF 249**	Gravimetry (Röse -Gottlieb)	I
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Water[14] (moisture)	**Described in Annex D***	**Gravimetry, drying at 102°C**	**IV**

Commodity	Provision	Method	Principle	Type
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Milk protein in MSNF[13]	ISO 5537 | IDF 26 and **ISO 23318 | IDF 249** and ISO 8968 - 1 | IDF 20 - 1	Calculation from total solids content, fat content and protein content Gravimetry, drying at 87°C and Gravimetry (Röse -Gottlieb) and Titrimetry (Kjeldahl)	IV
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	**Milk protein in MSNF[13]**	**Described in Annex D* and ISO 23318 | IDF 249 and ISO 8968-1 | IDF 20-1**	**Calculation from total solids content, fat content and protein content Gravimetry, drying at 102°C and Gravimetry (Röse-Gottlieb) and Titrimetry (Kjeldahl)**	**IV**
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Milk protein in MSNF[13]	ISO 5537 | IDF 26 and **ISO 23318 | IDF 249** and AOAC 991.20	Calculation from total solids content, fat content and protein content Gravimetry, drying at 87°C and Gravimetry (Röse -Gottlieb) and Titrimetry (Kjeldahl)	IV
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	**Milk protein in MSNF[13]**	**Described in Annex D* and ISO 23318 | IDF 249 and AOAC 991.20**	**Calculation from total solids content, fat content and protein content Gravimetry, drying at 102°C and Gravimetry (Röse -Gottlieb) and Titrimetry (Kjeldahl)**	**IV**

Commodity	Provision	Method	Principle	Type
Blend of sweetened condensed skimmed milk and vegetable fat	Total fat	**ISO 23318 | IDF 249**	Gravimetry (Röse -Gottlieb)	I
Blend of sweetened condensed skimmed milk and vegetable fat (for products sweetened with sucrose only)	Milk solids -not -fat[13] (MSNF)	ISO 6734 | IDF 15 and **ISO 23318 | IDF 249** and ISO 2911 | IDF 35	Calculation from total solids content, fat content and sucrose content Gravimetry, drying at 102°C and Gravimetry (Röse -Gottlieb) and Polarimetry	IV
Blend of sweetened condensed skimmed milk and vegetable fat (for products sweetened with sucrose only)	Milk protein in MSNF[13]	ISO 6734 | IDF 15 and **ISO 23318 | IDF 249** and ISO 2911 | IDF 35 and ISO 8968 -1 | IDF 20 - 1	Calculation from total solids content, fat content, sucrose content and protein content Gravimetry, drying at 102°C and Gravimetry (Röse -Gottlieb) and Polarimetry and Titrimetry (Kjeldahl)	IV
Blend of sweetened condensed skimmed milk and vegetable fat (for products sweetened with sucrose only)	Milk protein in MSNF[13]	ISO 6734 | IDF 15 and **ISO 23318 | IDF 249** and ISO 2911 | IDF 35 and AOAC 991.20	Calculation from total solids content, fat content, sucrose content and protein content Gravimetry, drying at 102°C and Gravimetry (Röse-Gottlieb) and Polarimetry and Titrimetry (Kjeldahl)	IV
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat	Total fat	**ISO 23318 | IDF 249**	Gravimetry (Röse -Gottlieb)	I

Commodity	Provision	Method	Principle	Type
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat (for products sweetened with sucrose only)	Milk solids -not -fat[13] (MSNF)	ISO 6734 | IDF 15 and **ISO 23318 | IDF 249** and ISO 2911 | IDF 35	Calculation from total solids content, fat content and sucrose content Gravimetry, drying at 102°C and Gravimetry (Röse -Gottlieb) and Polarimetry	IV
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat (for products sweetened with sucrose only)	Milk protein in MSNF[13]	ISO 6734 | IDF 15 and **ISO 23318 | IDF 249** and ISO 2911 | IDF 35 and ISO 8968 -1|IDF 20 - 1	Calculation from total solids content, fat content, sucrose content and protein content Gravimetry, drying at 102°C and Gravimetry (Röse - Gottlieb) and Polarimetry and Titrimetry (Kjeldahl)	IV
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat (for products sweetened with sucrose only)	Milk protein in MSNF[13]	ISO 6734 | IDF 15 and **ISO 23318 | IDF 249** and ISO 2911 | IDF 35 and AOAC 991.20	Calculation from total solids content, fat content, sucrose content and protein content Gravimetry, drying at 102°C and Gravimetry (Röse-Gottlieb) and Polarimetry and Titrimetry (Kjeldahl)	IV
Cheese	Milkfat	**ISO 23319 | IDF 250**	Gravimetry (Schmid-Bondzynski - Ratzlaff)	I
Cheeses, individual	Milkfat in dry matter	ISO 5534 | IDF 4 **ISO 23319 | IDF 250**	Calculation from dry matter content and fat content Gravimetry, drying at 102°C and Gravimetry	I
Cheeses in brine	Milkfat in dry matter (FDM)	ISO 5534 | IDF 4 **ISO 23319 | IDF 250**	Calculation from dry matter content and fat content Gravimetry, drying at 102°C and Gravimetry (Schmid- BondzynskiRatzlaff)	I

Commodity	Provision	Method	Principle	Type
Cottage cheese	Fat-free dry matter	ISO 5534 | IDF 4 and **ISO 23319 | IDF 250**	Calculation from dry matter content and fat content Gravimetry, drying at 102°C Gravimetry (Schmid-Bondzynski-Ratzlaff)	I
Cottage cheese (for samples containing lactose up to 5%)	Milkfat in dry matter	ISO 5534 | IDF 4 and **ISO 23319 | IDF 250**	Calculation from dry matter content and fat content Gravimetry, drying at 102°C and Gravimetry (Schmid-Bondzynski-Ratzlaff)	I
Cottage cheese (for samples containing lactose up to 5%)	Milkfat	**ISO 23319 | IDF 250**	Gravimetry (Schmid-Bondzynski-Ratzlaff)	I
Cream cheese	Moisture on fat-free basis	ISO 5534 | IDF 4 **ISO 23319 | IDF 250**	Calculation from fat content and moisture content Gravimetry drying at 102°C (forced air oven) Gravimetry (Schmid-Bondzynski-Ratzlaff)	I
Dairy permeate powders	Milkfat	**ISO 23318 | IDF 249**	Gravimetry (Röse-Gottlieb)	I
Edible casein products	Milkfat (Total fat)	**ISO 23319 | IDF 250**	Gravimetry (Schmid-Bondzynski-Ratzlaff)	I
Milk powders and cream powders	**Water[14] (Moisture)**	**Described in Annex D***	**Gravimetry, drying at 102°C**	**IV**
Milk powders and cream powders	Milkfat	**ISO 23318 | IDF 249**	Gravimetry (Röse-Gottlieb)	I
Mozzarella	Milkfat in dry matter – with high moisture	ISO 5534 | IDF 4 and **ISO 23319 | IDF 250**	Calculation from dry matter content and fat content Gravimetry, drying at 102°C and Gravimetry (Schmid-Bondzynski-Ratzlaff)	I

Commodity	Provision	Method	Principle	Type
Mozzarella	Milkfat in dry matter – with low moisture	ISO 5534 | IDF 4 and **ISO 23319 | IDF 250**	Calculation from dry matter content and fat content Gravimetry, drying at 102°C and Gravimetry (Schmid-Bondzynski-Ratzlaff)	I
Whey cheeses by coagulation	Milkfat	**ISO 23319 | IDF 250**	Gravimetry (Schmid-Bondzynski-Ratzlaff)	I
Whey cheeses by coagulation	Milkfat in dry matter	**ISO 23319 | IDF 250** and ISO 5534 | IDF 4	Calculation from fat content and dry matter content Gravimetry (Schmid-Bondzynski-Ratzlaff) Gravimetry, drying at 102°C	I
Fermented milks	Milkfat	**ISO 23318 | IDF 249**	Gravimetry (Röse-Gottlieb)	I
Cream	Milkfat	**ISO 23318 | IDF 249**	Gravimetry (Röse-Gottlieb)	I
Creams lowered in milkfat content	Milkfat	**ISO 23318 | IDF 249** / AOAC 995.19	Gravimetry (Röse-Gottlieb)	I
Evaporated milks	Milkfat	**ISO 23318 | IDF 249**	Gravimetry (Röse-Gottlieb)	I
Evaporated milks	Milk Protein in MSNF[13]	ISO 6731 | IDF 21 and **ISO 23318 | IDF 249** and ISO 8968-1 | IDF 20-1	Calculation from total solids content, fat content and protein content Gravimetry, drying at 102°C and Gravimetry (Röse-Gottlieb) and Titrimetry (Kjeldahl)	I
Sweetened condensed milk	Milkfat	**ISO 23318 | IDF 249**	Gravimetry (Röse-Gottlieb)	I

Commodity	Provision	Method	Principle	Type
Sweetened condensed milks (for products sweetened with sucrose only)	Milk Protein in MNSF[13]	ISO 6734 | IDF 15 and **ISO 23318 | IDF 249** and ISO 2911 | IDF 35 and ISO 8968-1 | IDF 20-1	Calculation from total solids content, fat content, sucrose, and protein content Gravimetry, drying at 102°C and Polarimetry Gravimetry (Röse-Gottlieb) Titrimetry (Kjeldahl)	I
Whey cheeses by concentration (carbohydrate contents below 5%)	Milkfat (Total fat)	**ISO 23318 | IDF 249**	Gravimetry (Röse Gottlieb)	I
Whey cheeses by concentration (for carbohydrate content under 5%)	Milkfat in dry matter (total fat in dry matter)	**ISO 23318 | IDF 249** and ISO 2920 | IDF 58	Calculation from fat content and dry matter content Gravimetry (Röse Gottlieb) Gravimetry, drying at 88°C	I
Whey powders	Milkfat	**ISO 23318 | IDF 249**	Gravimetry (Röse-Gottlieb)	I
Infant formula	Total fat	AOAC 989.05 **ISO 23318 | IDF 249**	Gravimetry (Röse-Gottlieb)	I

***Due to accessibility to equipment and calibration of the method ISO 5537 | IDF 26, the method described in the Annex is listed as Type IV**

ANNEX D – Test moisture method at normal pressure (102 ± 2)°C

Products	Parameter
Powdered milk, Powdered cream, and Blend of skimmed milk powder with vegetable fat	Moisture

DESCRIPTION OF THE METHOD: DETERMINATION OF MOISTURE

1. SCOPE

This Standard specifies a method for the determination of moisture content for all types of powdered milk, powdered cream, and mixtures of powdered skimmed milk with vegetable fat.

2. DEFINITION

The content is the mass loss determined by the procedure specified in this Standard. It is expressed in percentage by mass g/100 g.

3. PRINCIPLE

A portion of the sample is dried in an oven set at (102 ± 2) °C until constant weight and weighed to determine the loss of mass.

4. EQUIPMENT

Common laboratory equipment and, in particular, the following.

4.1 Analytical balance, capable of weighing with a precision of 1 mg, with a minimum resolution of 0.1 mg.

4.2 Drying oven, with good ventilation, as far as possible with forced ventilation, capable of being thermostatically maintained at (102 ± 2) °C throughout the workspace, with a temperature controller.

4.3 Desiccator, with freshly dried silica gel with hygrometric indicator or another effective desiccant.

4.4 Flat-bottomed dishes, approximately 25 mm deep, approximately 50 mm in diameter, and made of an appropriate material (for example, glass, stainless steel, nickel, or aluminium), fitted with tight-fitting, removable lids easily.

5. SAMPLING

It is important that the laboratory receive a truly representative sample and that it has not been damaged or changed during transport or storage.

Sampling is not part of the method specified in this Standard. A recommended sampling method is provided in ISO 707 | IDF 50.

6. TEST SAMPLE PREPARATION

Transfer the entire sample to a dry, tightly closed container with a capacity of approximately twice the volume of the sample. Mix thoroughly by turning and shaking the container.

7. PROCEDURE

7.1 Preparation of the dish

7.1.1 Heat the uncovered capsule and its lid (4.4) in the oven (4.2) controlled at (102 ± 2)°C, for 1 h.

7.1.2 Transfer the capped dish to the desiccator (4.3), allow it to cool to room temperature in the balance room, and weigh (4.1) to the nearest 0.1 mg.

7.2 Test sample

7.2.1 Place 1 - 1.5 g of the prepared test sample (6) in the dish, cover with the lid and weigh to the nearest 0.1 mg.

7.3 Determination

7.3.1 Uncover the capsule and place it together with the lid in the oven (4.2), controlled at (102 ± 2)°C for 2 hrs.

7.3.2 Replace the cap, transfer the capped dish to the desiccator, allow to cool to balance room temperature, and weigh to the nearest 0.1 mg.

7.3.3 Uncover the capsule and heat again, along with its lid, on the oven for 1 h. Then repeat operation 7.3.2.

7.3.4 Repeat this process until the difference in mass between two successive weighings does not exceed 0.5 mg. Record the lowest mass.

8. CALCULATION AND EXPRESSION OF RESULTS

8.1 Calculation

The moisture content in the sample, expressed in g/100 g, is equal to:

$$\text{moisture} = \frac{(m_1 - m_2) \times 100}{(m_1 - m_0)}$$

where,

m_0 is the mass, in grams, of the dish and lid (7.1.2)

m_1 is the mass, in grams, of the dish, lid and test sample before drying (7.2.1)

m_2 is the mass, in grams, of the dish, lid and test sample after drying (7.3.4)

8.2 Expression of test results

Express the sample results to two decimal places.

1.7 FATS AND OILS

Commodity	Provision	Method	Principle	Type
Fats and oils	**Synthetic phenolic antioxidants**	**~~AOCS Ce 6-86~~ AOCS Ce 6a-2021**	Liquid chromatography	II
Fats and oils	**Synthetic phenolic antioxidants**	AOAC 983.15	Liquid chromatography	**III**
Fish oils	Fatty acid composition	**AOCS ~~Ce 2b-11~~ Ce 2c-66 and AOCS Ce 1i-07 /** AOCS Ce 1j-07	**Preparation of methyl esters and Gas Chromatography**	III
Fish oils	Fatty acid composition	**ISO 12966-2 and ISO 12966-4**	**Preparation of methyl esters and Gas Chromatography**	III
Named Animal Fats	Fatty acid composition	**AOCS Ce 2-66 and Ce 1j-07**	**Preparation of methyl esters and Gas Chromatography**	**II**
Named Animal Fats	Fatty acid composition	**ISO 12966-2 and ISO 12966-4**	**Preparation of methyl esters and Gas Chromatography**	**~~II~~ III**
Named Animal Fats	Titre	**ISO 935**	Thermometry	I
Named Animal Fats	Titre	**AOCS Cc 12-59[a]**	Thermometry	**~~I~~ IV**
Named Vegetable Oils	Crismer value	AOCS Cb 4-35 and AOCS Ca 5a-40	Calculation from individual fatty acid composition (gas chromatography of methyl esters) and turbidity	I
Named Vegetable Oils	Halphen test	AOCS Cb 1-25	Colorimetry	I
Named Vegetable Oils	Unsaponifiable matter	**ISO 3596 / AOCS Ca 6b-53**	**Diethyl ether extraction and** gravimetry, drying at 103°C and titrimetry (colorimetry) **and correction for free fatty acids titrimetry (colorimetry)[c]**	I

Commodity	Provision	Method	Principle	Type
Named Vegetable Oils	Unsaponifiable matter	**ISO 18609[b]**	**Hexane extraction and** Gravimetry, drying at 103°C and titrimetry (colorimetry) **and correction for free fatty acids titrimetry (colorimetry)[c]**	~~I~~ **IV**
Fish Oil	**Vitamin A[d]**	European Parharmcopeia Monograph on Cod Liver Oil (Type A), monograph 01/2005:1192, with LC end-point 2.2.29	~~LC~~ **Liquid Chromatography**	III
Fish Oil	**Vitamin A[d]**	EN 12823-1 ~~**(Determination of vitamin A by high performance liquid chromatograph – Part 1: Measurement of all-E-retinol and 13-Z-retinol)**~~	~~LC~~ **Liquid Chromatography**	~~III~~ **II**
Fish Oil	Vitamin D[e]	NMKL 167 / EN 12821 ~~**(Determination of vitamin D by high performance liquid chromatography – Measurement of cholecalciferol (D3) or ergocalciferol (D2))**~~	~~LC~~ **Liquid Chromatograpy**	~~III~~ **II**

[a] AOCS Cc 12-59 is the preferred method in certain regions. Due to difference in practical application of AOCS Cc 12-59 compared to ISO 935, it is listed as a Type IV method

[b] results obtained from ISO 18609 are systematically lower. In case of limitations due to climate or regulations that prohibit the use of diethyl ether, ISO 18609 can be used instead of the Type I method

[c] The technique to ISO 18609 is gravimetric. The correction by titration and colorimetry is only when it is necessary to correct for free fatty acids

[d]The respective standard on fish oils CXS 329-2017 states that Vitamin A is expressed as 'Retinol equivalents'(RE) where RE takes into account the fact that different vitamers of vitamin A differ in activity. ISO/TR 23304:2021 "Food products – Guidance on how to express vitamins and their vitamers" may give clarity on this matter, for example for the relevant activities of the all-E-retinol levels and 13-Z-retinol levels.

[e] The provision accounts for Vitamin D2 and D3.

1.8 CEREALS, PULSES AND LEGUMES

Commodity	Provision	Method	Principle	Type
Certain pulses **(Soybeans)**	Moisture	ISO 665	Gravimetry **(oven drying at 103ºC**	I
Certain pulses **except soybeans**	Moisture	**ISO 24557 / AACC 44-17.01**	Gravimetry **(oven drying at 130ºC)**	I
Degermed maize (corn) meal and maize (corn) grits	Fat, crude	AOAC 945.38F~~;~~ **and** 920.39C **and ICC 110/1**	Calculation from moisture and Gravimetry (ether extraction)	I
Degermed maize (corn) meal and maize (corn) grits	Moisture	**ICC 110/1**	Gravimetry **(oven drying at 130 – 133ºC)**	I
Degermed maize (corn) meal and maize (corn) grits	Particle size (granularity)	AOAC 965.22[1]	Gravimetry (Sieving)	I
Degermed maize (corn) meal and maize (corn) grits	Protein	ICC 105/**2 and ICC 110/1**	Calculation from moisture and Titrimetry (Kjeldahl digestion)	I
Durum wheat semolina and durum wheat flour	Ash ~~(semolina)~~	AOAC 923.03 **/** ISO 2171 and ISO 712 **/** ICC 110/1	Calculation from moisture and Gravimetry **(incineration at 550ºC)**	I
Durum wheat semolina and durum wheat flour	Moisture	ISO 712 **/** ICC 110/1	Gravimetry **(oven drying at 130 – 133ºC)**	I
Durum wheat semolina and durum wheat flour	Protein ~~(N x 5.7)~~	ICC 105/~~1~~**2 and ISO 712 / ICC 110/1**	Calculation from moisture and Titrimetry (Kjeldahl digestion)	I
Instant Noodles	Acid Value	described in the standard, will be moved to 234	Titrimetry (ether **extraction**)	I
Instant Noodles	Moisture	described in the standard, will be moved to 234	Gravimetry **(oven drying at 105ºC)**	I

Commodity	Provision	Method	Principle	Type
Maize (corn)	Moisture	ISO 6540 **/ ICC 110/1**	Gravimetry **(oven drying at 130 – 133°C)**	I
Pearl millet flour	Ash	AOAC 923.03 **/ ISO 2171** and **ISO 712 / ICC 110/1**	**Calculation from moisture and Gravimetry (incineration at 550°C)**	I
Pearl millet flour	Fat, crude	AOAC 945.38F~~;~~ **and** 920.39C **and ISO 712 / ICC 110/1**	**Calculation from moisture and** Gravimetry (ether extraction)	I
Pearl millet flour	Fibre, crude	ISO 5498 ~~(B.5 Separation)~~ **and ISO 712 / ICC 110/1**	**Calculation from moisture and** Gravimetry (**extraction and filtration**)	I
Pearl millet flour	Moisture	ISO 712 ~~;~~**/** ICC 110/1	Gravimetry **(oven drying at 130 – 133°C)**	I
Pearl millet flour	Protein	**ISO 20483 and ISO 712 / ICC 110/1**	**Calculation from moisture and Titrimetry (Kjeldahl digestion)**	I
Sorghum flour	Fat, crude	AOAC 945.38F~~;~~ **and** 920.39C **and ISO 712 / ICC 110/1**	**Calculation from moisture and** Gravimetry (ether extraction)	I
Sorghum flour	Fibre, crude	ICC 113 **/** ISO 6541 **and ISO 712 / ICC 110/1**	**Calculation from moisture and Gravimetry (separation, incineration)**	I
Sorghum flour	Moisture	ISO 712 **/** ICC 110/1	Gravimetry **(oven drying at 130 – 133°C)**	I
Sorghum flour	Particle size (granularity)	AOAC 965.22[1]	Gravimetry (Sieving)	I

Commodity	Provision	Method	Principle	Type
Sorghum flour	Protein	ICC 105/1~~1~~**2 and ISO 712 / ICC 110/1**	**Calculation from moisture and** Titrimetry (Kjeldahl digestion)	I
Sorghum flour	Tannins	ISO 9648 **and ISO 712 / ICC 110/1**	**Calculation from moisture and** Spectrophotometry	I
Sorghum grains	Fat, crude	AOAC 945.38F~~,~~ **and** 920.39C **and ISO 6540**	**Calculation from moisture and** Gravimetry (ether extraction)	I
Sorghum grains	Moisture	ISO 6540	Gravimetry **(oven drying at 130 – 133ºC)**	I
Sorghum grains	Protein	ICC 105/1~~1~~**2 and ISO 6540**	Calculation from moisture and Titrimetry (Kjeldahl digestion)	I
Sorghum grains	Tannins	ISO 9648 **and ISO 6540**	Calculation from moisture and Spectrophotometry	I
Soy protein products	Ash	AOAC 923.03 **/** ISO 2171~~:~~ ~~(Method B)~~ and AOAC 925.09	Calculation from moisture and Gravimetry **(incineration at 550ºC)**	I
Soy protein products	Fibre, crude	ISO 5498 **and AOAC 925.09**	**Calculation from moisture and** Gravimetry ~~(separation)~~ **(extraction and filtration)**	I
Soy protein products	Moisture	AOAC 925.09	Gravimetry (vacuum oven **at 98 – 100ºC**)	I
Vegetable protein products	Ash	AOAC 923.03 **/** ISO 2171 ~~(Method B)~~ **and AOAC 925.09**	**Calculation from moisture and** Gravimetry **(incineration at 550ºC)**	I

Commodity	Provision	Method	Principle	Type
Vegetable protein products	Fibre, crude	AACC ~~32-17~~ 32-10.01 **and AOAC 925.09**	**Calculation from moisture and Gravimetry** (Ceramic fibre filtration)	I
Vegetable protein products	Moisture	AOAC 925.09	Gravimetry (vacuum oven **at 98 – 100ºC**)	I
Wheat flour	Fat acidity	**ISO 7305 and ISO 712 / ICC 110/1**	**Calculation from moisture and** Titrimetry **(extraction)**	I
Wheat flour	Moisture	ISO 712 **/** ICC 110/1	Gravimetry **(oven drying at 130 – 133ºC)**	I
Wheat flour	Particle size (granularity)	AOAC 965.22[1]	Gravimetry (Sieving)	I
Wheat flour	Protein	ICC 105/~~1~~**2 and ISO 712÷ / ICC 110/1**	Calculation from moisture and Titrimetry (Kjeldahl digestion)	I
Wheat protein products including wheat gluten	Fibre, crude[1]	AOAC 962.09 **and AOAC 925.09**	Calculation from moisture and **Gravimetry** ~~Ceramic fibre~~ (**ceramic fibre filtration)**	I
Wheat protein products including wheat gluten	**Moisture**	**AOAC 925.09**	**Gravimetry (vacuum oven at 98 – 100°C)**	**I**
Wheat protein products including wheat gluten	Crude Protein[1]~~;~~ excluding added vitamins, minerals, amino acids, and optional ingredients	Vital wheat gluten and devitalized wheat gluten **ISO 20483 and AOAC 925.09**	**Calculation from moisture and** Titrimetry (Kjeldahl digestion)	I
		Solubilized wheat protein **ISO 20483 and AOAC 925.09**	Calculation from moisture and ~~Kjeldahl~~ Titrimetry (Kjeldahl digestion) (~~wheat protein in flour N x 5.7~~)	I

Commodity	Provision	Method	Principle	Type
Wheat protein products including wheat gluten	Ash	AOAC 923.03 **/** ISO 2171~~:~~ ~~method B~~ and AOAC 925.09	Calculation from moisture and Gravimetry **(incineration at 550ºC)**	I
Whole and decorticated pearl millet grains	Ash	AOAC 923.03 **/ ISO 2171** and ISO 712 **/** ICC 110/1	Calculation from moisture and Gravimetry **(incineration at 550ºC)**	I
Whole and decorticated pearl millet grains	Fat, crude	AOAC 945.38F~~;~~ **and** 920.39C **and ISO 712 / ICC 110/1**	Calculation from moisture and Gravimetry (ether extraction)	I
Whole and decorticated pearl millet grains	Fibre, crude	ISO 5498 ~~(B.5 separation)~~ and ISO 712 **/** ICC 110/1	Calculation from moisture and Gravimetry **(filtration through filter paper)**	I
Whole and decorticated pearl millet grains	Moisture	ISO 712 **/** ICC 110/1	Gravimetry **(oven drying 130 – 133ºC)**	I
Whole and decorticated pearl millet grains	Protein	**ISO** 20483 **and ISO 712 / ICC 110/1**	**Calculation from moisture and** Titrimetry (Kjeldahl-digestion)	I
Whole maize (corn) meal	~~Crude~~ Fat, crude	AOAC 945.38F~~;~~ **and** 920.39C **and ICC 110/1**	Calculation from moisture and Gravimetry (ether extraction)	I
Whole maize (corn) meal	Moisture	ICC 110/1 **/ ISO 6540**	Gravimetry **(oven drying 130 – 133ºC)**	I
Whole maize (corn) meal	Particle size (granularity)	AOAC 965.22[1] ~~and ISO 3310-1~~	Gravimetry (Sieving)	I
Whole maize (corn) meal	Protein	ICC 105/~~1~~**2 and ICC 110/1**	**Calculation from moisture and** Titrimetry (Kjeldahl digestion)	I

Commodity	Provision	Method	Principle	Type
Gari	**Total acidity**	**ISO/~~DP~~ 7305 and ISO 712**	**Titrimetry (ethanol extraction)**	**I**
Gari	Crude fibre	ISO 5498 **and ISO 712**	Gravimetry (separation)	I
Gari	Ash	ISO 2171 **and ISO 712**	Calculation from moisture Gravimetry (incineration **at 550°C**)	I
Gari	Moisture	ISO 712	Gravimetry **(oven drying 130 – 133°C)**	I
Edible Cassava flour	Moisture	ISO 712	Gravimetry **(oven drying at 98 – 100˚C)**	**I**
Edible Cassava flour	Crude fibre	ISO 5498 ~~(B.5 separation)~~	Gravimetry (separation)	I
Edible Cassava flour	**Ash**	**ISO 2171 and ISO 712**	**Calculation from moisture** **Gravimetry** **(incineration at 550°C)**	**I**

[1]sieve specifications as in ISO 3310/1

1.9 PROCESSED FRUITS AND VEGETABLES

Commodity	Provision	Method	Principle	Type
Processed fruits and vegetables* **(Jams, Jellies, Marmalades, pickled cucumbers, mango chutney, Coconut Milk and Coconut Cream)**	Benzoic acid	NMKL 124	Liquid Chromatography **(UV)**	II
Processed fruits and vegetables***** **(Jams, Jellies, Marmalades, pickled cucumbers, mango chutney, Coconut Milk and Coconut Cream)**	Benzoic acid	AOAC 983.16	Gas Chromatography **(Flame ionization)**	III
Processed fruits and vegetables***** **(Canned strawberries, pickled cucumbers, preserved tomatoes, canned citrus fruits, certain canned vegetables)**	Calcium	AOAC 968.31	Complexometry / Titrimetry	II
Processed fruits and vegetables	Drained Weight	AOAC 968.30 (Codex General Method)	~~Sieving~~ Gravimetry **(Sieving)**	I
Processed fruits and vegetables	Fill of **glass** containers	**ISO 8106**	**Gravimetry**	I
Processed fruits and vegetables	**Fill of metal containers**	**ISO 90-1**	**Gravimetry**	I
Processed fruits and Vegetables* **(Pickled cucumbers, table olives, processed tomato concentrates, preserved tomatoes, mango chutney, and aqueous coconut products)** ~~except canned bamboo shoots, pH determined by AOAC 981.12)~~	pH	ISO 1842	Potentiometry	IV
Canned bamboo shoots	**pH**	**AOAC 981.12**	**Potentiometry**	**IV**

Commodity	Provision	Method	Principle	Type
Processed fruits and vegetables* **(Pickled cucumbers, table olives, processed tomato concentrates, preserved tomatoes, mango chutney, and aqueous coconut products)**	pH	AOAC 981.12	Potentiometry	III
Processed fruits and vegetables* **(Pickled cucumbers, table olives, processed tomato concentrates, preserved tomatoes, mango chutney, and aqueous coconut products)**	pH	NMKL 179	Potentiometry	II
Processed fruits and vegetables* **(Pickled cucumbers, processed tomato concentrates, preserved tomatoes, canned applesauce, jams, jellies and marmalades, mango chutney, and certain canned fruit)**	Soluble solids **(packing medium)**	ISO 2173	Refractometry	I
Processed fruits and vegetables* **(Jams, Jellies, Marmalades, pickled cucumbers)**	Sorbates	AOAC 983.16	Gas Chromatography **(Flame ionization)**	III
Processed fruits and vegetables* **(Jams, Jellies, Marmalades, pickled cucumbers)**	Sorbates	NMKL 124	Liquid Chromatography **(UV)**	II
Processed fruits and vegetables	Tin	AOAC 980.19 (Codex general method)	~~AAS~~ **Atomic Absorption Spectrophotometry (Flame)**	II
Processed fruits and vegetables	Total solids	AOAC 920.151	Gravimetry	I
Aqueous Coconut Products	Total Fats	**ISO 23318 | IDF 249**	Gravimetry (Röse-Gottlieb)	I
Aqueous Coconut Products	Total solids	ISO 6731 | IDF 21	Gravimetry	I

Commodity	Provision	Method	Principle	Type
Aqueous Coconut Products	Non-fat solids	**ISO 23318 | IDF 249** and ISO 6731 | IDF 21	Calculation: Gravimetry (Röse-Gottlieb) Gravimetry	I
Aqueous Coconut Products	Moisture	ISO 6731 | IDF 21	~~Calculation:~~ Gravimetry	I
Canned Apple Sauce	Fill of **glass** containers	**ISO 8106**	~~Weighing~~ **Gravimetry**	I
Canned Apple Sauce	**Fill of metal containers**	**ISO 90-1**	~~Weighing~~ **Gravimetry**	I
Canned Apple Sauce	Soluble solids **(packing medium)**	ISO 2173 (Codex general method for processed fruits and vegetables)	Refractometry	I
Canned green beans and wax beans	Tough Strings	CAC/RM 39	Stretching	I
Canned green peas	**Fill of glass containers**	**ISO 8106**	**Gravimetry**	I
Canned green peas	**Fill of metal containers**	**ISO 90-1**	**Gravimetry**	I
Canned green peas	Types of peas, distinguishing	CAC/RM 48	Visual ~~inspection~~ **examination**	I
Canned mangoes	**Soluble Solids (packing medium)**	AOAC 932.14C	Brix spindle method **(refractometry)**	I

Commodity	Provision	Method	Principle	Type
Canned mushrooms	~~Washed~~ Drained weight	**AOAC 968.30**	**Gravimetry** (Sieving)	I
Canned palmito	Mineral impurities	ISO 762	Gravimetry	I
Canned Stone Fruits	Drained weight	AOAC 968.30	Gravimetry **(sieving)**	I
Canned Stone Fruits	Soluble solids **(packing medium)**	**ISO 2173**	Refractometry	I
Canned strawberries	Calcium	AOAC 968.31	Complexometry / titrimetry	II
Canned strawberries	Mineral impurities	**ISO 762**	Gravimetry	I
Certain canned citrus fruits	Calcium	NMKL 153	**Atomic Absorption Spectrophotometry (Flame)**	II
Certain canned citrus fruits	Calcium	AOAC 968.31	Complexometry / Titrimetry	III
Citrus marmalade	Calcium	AOAC 968.31	Complexometry / titrimetry	II
Dates	Identification of defects	Described in **CXS 143**	Visual ~~inspection~~ **examination**	I
Dates	Moisture	AOAC 934.06	Gravimetry (vacuum oven)	I
Desiccated coconut	Total acidity of the extracted oil	**ISO 660 / AOCS Cd 3d-63**	Potenciometry / Titrimetry	I
Desiccated coconut	Ash	AOAC 950.49	Gravimetry **(Ashing)**	I

Commodity	Provision	Method	Principle	Type
Desiccated coconut	Extraneous vegetable matter	Described in ~~the Standard~~ **CXS 177**	Counting extraneous material with the naked eye	IV
Desiccated coconut	Moisture	AOAC 925.40	Gravimery (loss on drying)	I
Desiccated coconut	Oil content	AOAC 948.22	Gravimetry	I
Dried apricots	Identification of defects	Described in the Standard	Visual examination (gravimetry)	I
Dried apricots	Moisture	AOAC 934.06	Gravimetry (vacuum oven)	I
Dried apricots	Sulphur dioxide	AOAC 963.20	Colorimetry	II
~~Jams (fruit preserves) and jellies~~ **Jams, Jellies, and Marmalades**	Fill of **Glass** Containers	**ISO 8106**	**Gravimetry**	I
~~Jams (fruit preserves) and jellies~~ **Jams, Jellies, and Marmalades**	Soluble solids	ISO 2173	Refractometry	I
Mango chutney	Ash insoluble in HCl	ISO 763	Gravimetry	I
Pickled cucumbers	Acidity, total	AOAC 942.15	Titrimetry	I
Pickled cucumbers	Drained weight	AOAC 968.30	Gravimetry	I
Pickled cucumbers	Mineral impurities	**ISO 762**	Gravimetry	I
Pickled cucumbers	Salt ~~in brine~~ **(NaCl)**	AOAC 971.27 (Codex general method)	Potentiometry	II

Commodity	Provision	Method	Principle	Type
Pickled cucumbers	Volume fill by displacement	Described in the Standard	Displacement	I
Preserved tomatoes	Calcium	AOAC 968.31	Complexometry / titrimetry	III
Preserved tomatoes	Calcium	NMKL 153	Atomic Absorption Spectrophotometry **(Flame)**	II
Preserved tomatoes	~~Minimum~~ Drained Weight	AOAC 968.30	Gravimetry (sieving) note: Use a No. 14 screen instead of '7/16' or No. 8	I
Preserved tomatoes	Mould count	AOAC 965.41	Howard mould count	I
Processed tomato concentrates	Lactic acid	~~EN 2631~~ **EN 12631**	**Spectrometry** (Enzymatic determination)	II
Processed tomato concentrates	Mineral impurities (sand)	**ISO 762**	Gravimetry	**I**
Processed tomato concentrates	Mould count	AOAC 965.41	Howard mould count	I
Processed tomato concentrates	Sodium chloride	AOAC 971.27 (Codex general method)	Potentiometry	II
Processed tomato concentrates	Tomato soluble solids	AOAC 970.59	Refractometry	I
Raisins	Mineral impurities	**ISO 762**	**Gravimetry** (Ashing)	I
Raisins	Mineral oil	CAC/RM 52	Extraction and separation on alumina	II
Raisins	Moisture	AOAC 972.20	Electrical conductance	I

Commodity	Provision	Method	Principle	Type
Raisins	Sorbitol	AOAC 973.28	Gas chromatography **(flame ionization)**	II
Raisins	Sulphur dioxide	AOAC 963.20	Colorimetry	II
Table olives	Drained weight	AOAC 968.30 (Codex general method for processed fruits and vegetables)	~~Sieving~~ Gravimetry **(sieving)**	I
Table olives	Fill of glass containers	**ISO 8106**	**Gravimetry**	I
Table olives	**Fill of metal containers**	**ISO 90-1 (for metal containers) (Codex general method for processed fruits and vegetables)**	~~Weighing~~ **Gravimetry**	I
Table olives	pH of brine	NMKL 179 (Codex general method for processed fruits and vegetables)	Potentiometry	II
Table olives	pH of brine	AOAC 981.12 (Codex general method for processed fruits and vegetables)	Potentiometry	III
Table olives	pH of brine	ISO 1842	Potentiometry	IV
Table olives	Salt in brine	AOAC 971.27 | NMKL 178 (Codex general method)	Potentiometry	II
Table olives	Tin	NMKL 190 | EN 15764	**Atomic Absorption Spectrophotometry (Flame)** ~~AAS~~	II

PART 2

METHODS OF ANALYSIS FOR REVOCATION BY CAC46

2.1 CODEX COMMITTEE ON CONTAMINANTS IN FOODS (CCCF)

Commodity	*Provision*	*Method*	*Principle*	*Type*
Fats and Oils and Related Products				
Fats and Oils (all)	Lead	AOAC 994.02 / ISO 12193 / AOCS Ca 18c-91	Atomic absorption spectrophotometry (direct graphite furnace)	II
Named Vegetable Oils	Lead	AOAC 994.02 / ISO 12193 / AOCS Ca 18c-91	Atomic absorption spectrophotometry (direct graphite furnace)	II
Olive Oils and Olive Pomace Oils	Lead	AOAC 994.02 or ISO 12193 or AOCS Ca 18c-91	AAS	II
Butter	Lead	AOAC 972.25 (Codex general method)	Atomic absorption spectrophotometry	IV
Edible casein products	Lead	NMKL 139 (Codex general method) AOAC 999.11	Atomic absorption spectrophotometry	IV
Edible casein products	Lead	NMKL 161 / AOAC 999.10	Atomic absorption spectrophotometry	IV
Edible casein products	Lead	ISO/TS 6733 | IDF/RM 133	Spectrophotometry (1,5-diphenylthiocarbazone)	IV
Processed Fruits and Vegetables				
Table olives	Lead	AOAC 999.11 | NMKL 139 (Codex general method)	AAS (Flame absorption)	II
Miscellaneous Products				
Food grade salt	Lead	EuSalt/AS 015	ICP-OES	III
Food grade salt	Lead	EuSalt/AS 013	Atomic absorption spectrophotometry	IV

2.2 FATS AND OILS

Commodity	Provision	Method	Principle	Type
Fats and oils	Butylhydroxyanisole, butylhydroxytoluene, tert-butylhydroquinone, & propyl gallate	AOAC 983.15; or AOCS Ce 6-86	Liquid chromatography	II
Fish oils	Fatty acid composition	*AOCS Ce 1a-13*	Capillary GLC	III
Fish oils	Fatty acid composition	*AOCS Ce 2-66*	Preparation of methyl esters by fatty acids	III
Fish oils	Fatty acid composition	*AOCS Ce 2b-11*	Alkali hydrolysis	III
Fish oils	Fatty acid composition	*AOCS Ce 2b-11*	Gas Chromatography of methyl esters	III
Fish oils	Fatty acid composition	*AOCS Ce 1i-07*	Capillary GLC	III
Fish oils	Fatty acid composition	*ISO 12966-2*	Gas chromatography	III
Fish oils	Fatty acid composition	*ISO 5508*	Gas chromatography	III
Fish oils	Fatty acid composition	AOCS Ce 2-66 and AOCS Ce 1a-13	Gas Chromatography of methyl esters	III
Fish oils	Fatty acid composition	AOCS Ce 1b 89	GLC	III
Named Animal Fats	GLC ranges of fatty acid composition	ISO 5508 and ISO 12966-2; or AOCS Ce 2-66 and Ce 1e-91 or Ce 1f-96	Gas chromatography of methyl esters	II
Named Animal Fats	Titre	ISO 935; or AOCS Cc 12-59	Thermometry	I
Named Vegetable Oils	Unsaponifiable matter	ISO 3596; or ISO 18609; or AOCS Ca 6b-53	Gravimetry	I

2.3 CEREALS, PULSES AND LEGUMES

Commodity	Provision	Method	Principle	Type
Degermed maize (corn) meal and maize (corn) grits	Moisture	ISO 712	Gravimetry	I
Instant Noodles	Extraction of oil from instant noodles	described in the standard	Gravimetry (ether extraction)	I
Pearl millet flour	Protein	AOAC 920.87	Calculation from moisture and-Titrimetry (Kjeldahl digestion)	I
Sorghum flour	Protein[1] (N x 6.25)	ISO 1871	Titrimetry (Kjeldahl digestion)	I
Sorghum grains	Protein[1] (N x 6.25)	ISO 1871	Titrimetry (Kjeldahl digestion)	I
Wheat flour	Protein[1] (N x 5.7)	ISO 1871	Titrimetry (Kjeldahl digestion)	I
Whole and decorticated pearl millet grains	Protein	AOAC 920.87	Titrimetry (Kjeldahl-digestion)	I
Wheat flour	Fat acidity	AOAC 939.05	Titrimetry	I
Wheat protein products including wheat gluten	Crude Protein[1]; excluding added vitamins, minerals, amino acids and optional ingredients	AOAC 979.09 (wheat protein in grain N x 5.7)	Kjeldahl	I
		AOAC 920.87 (wheat protein in flour N x 5.7)	Kjeldahl	I
Whole and decorticated pearl millet grains	Protein	AOAC 920.87	Titrimetry, Kjeldahl digestion	I
Whole maize (corn) meal	Moisture	ISO 712	Gravimetry	I
Gari	Moisture	ICC 109/1	Gravimetry	I

Commodity	Provision	Method	Principle	Type
Peanuts (raw)	Aflatoxins, total	AOAC 991.31	Immunoaffinity column (Aflatest)	II
Peanuts (raw)	Aflatoxins, total	AOAC 993.17	Thin layer chromatography	III
Peanuts (intended for further processing)	Aflatoxins, total	AOAC 975.36	Romer minicolumn	III
Peanuts (Cereals, shell-fruits and derived products (including peanuts))	Sum of aflatoxins B1, B2, G1 and G2	EN 12955 ISO 16050	HPLC with post column derivatization and immunoaffinity column clean up	III
Peanuts (intended for further processing	Aflatoxins, total	AOAC 979.18	Holaday-Velasco minicolumn	III

REVOCATION OF METHODS IN SPECIFIC COMMODITY STANDARDS

Commodity	Provision	Method	Notes
Sorghum grains	Fat	ISO 5986	See CXS 172
Sorghum grains	Fibre, crude	ICC 113 and ISO 6541	See CXS 172
Whole and decorticated pearl millet grains	Fat, crude	ISO 5986	See CXS 169
Whole maize (corn) meal	Fat, crude	ISO 5986	See CXS 154
Gari	Total acidity	AOAC method 1975 14.064 – 14.065	See CXS 151

2.4 PROCESSED FRUITS AND VEGETABLES

Commodity	Provision	Method	Principle	Type
Processed fruits and vegetables	Soluble solids	AOAC 932.12	Refractometry	I
Processed fruits and vegetables	Sorbates	NMKL 103	Gas Chromatography	III
Aqueous Coconut Products	Total Fats	ISO 1211 | IDF 1	Gravimetry (Röse-Gottlieb)	I
Aqueous Coconut Products	Non-fat solids	ISO 1211 | IDF 1	Calculation: Gravimetry (Röse-Gottlieb) Gravimetry	I
Canned Apple Sauce	Fill of containers	CAC/RM 46* (for glass containers) (Codex general method for processed fruits and vegetables) and ISO 90-1 (for metal containers) (Codex general method for processed fruits and vegetables)	Weighing	I
Canned Apple Sauce	Soluble solids	AOAC 932.12	Refractometry	I
Canned green peas	Proper fill (in lieu of drained weight)	CAC/RM 45	Pouring and measuring	I
Canned mushrooms	Washed Drained weight	CAC/RM 44	Sieving	I
Canned Stone Fruits	Drained weight	ISO:2173	Gravimetry	I
Canned Stone Fruits	Soluble solids	AOAC 932.14C	Refractometry	I
Canned strawberries	Mineral impurities	AOAC 971.33	Gravimetry	I

Commodity	Provision	Method	Principle	Type
Certain Canned Vegetables (palmito)	Mineral impurities (sand)	AOAC 971.33 ISO 762	Gravimetry	I
Jams (fruit preserves) and jellies	Fill of _Containers	CAC/RM 46	Weighing	I
Jams (fruit preserves) and jellies	Soluble solids	AOAC 932.12	Refractometry	I
Pickled cucumbers	Mineral impurities	AOAC 971.33	Gravimetry	I
Processed tomato concentrates	Mineral impurities (sand)	AOAC 971.33	Gravimetry	IV
Raisins	Mineral impurities	CAC/RM 51	Ashing	I
Table olives	Fill of containers	CAC/RM 46* (for glass containers) (Codex general method for processed fruits and vegetables) and ISO 90-1 (for metal containers) (Codex general method for processed fruits and vegetables)	Weighing	I

5.1 MILK AND MILK PRODUCTS

Commodity	Provision	Method	Principle	Type
Reduced fat blend of evaporated skimmed milk and vegetable fat / Blend of evaporated skimmed milk and vegetable fat / Blend of evaporated skimmed milk and vegetable fat /	Milk protein in MSNF[13]	ISO 1737 | IDF 13	Calculation from total solids content, fat content and protein content Gravimetry, drying at 102°C and Gravimetry (Röse-Gottlieb) and Titrimetry (Kjeldahl)	IV
Blend of evaporated skimmed milk and vegetable fat	Milk solids-not-fat[13] (MSNF)	ISO 1737 | IDF 13	Calculation from total solids content and fat content Gravimetry, drying at 102°C and Gravimetry (Röse-Gottlieb)	I
Reduced fat blend of evaporated skimmed milk and vegetable fat	Total fat	ISO 1737| IDF 13	Gravimetry (Röse-Gottlieb)	I
Reduced fat blend of evaporated skimmed milk and vegetable fat	Milk solids-not-fat (MSNF) [13]	ISO 1737 | IDF 13	Calculation from total solids content and fat content Gravimetry, drying at 102°C and Gravimetry (Röse-Gottlieb)	I
Blend of skimmed milk and vegetable fat in powdered form	Total fat	ISO 1736 | IDF 9	Gravimetry (Röse-Gottlieb)	I
Blend of skimmed milk and vegetable fat in powdered form	Milk protein in MSNF[13]	ISO 1736 | IDF 9	Calculation from total solids content, fat content and protein content Gravimetry, drying at 87°C and Gravimetry (Röse-Gottlieb) and Titrimetry (Kjeldahl)	IV

Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Total fat	ISO 1736 | IDF 9	Gravimetry (Röse -Gottlieb)	I
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Milk protein in MSNF[13]	ISO 1736 | IDF 9	Calculation from total solids content, fat content and protein content Gravimetry, drying at 87°C and Gravimetry (Röse -Gottlieb) and Titrimetry (Kjeldahl)	IV
Blend of sweetened condensed skimmed milk and vegetable fat	Total fat	ISO 1737 | IDF 13	Gravimetry (Röse -Gottlieb)	I
Blend of sweetened condensed skimmed milk and vegetable fat (for products sweetened with sucrose only)	Milk solids -not -fat[13] (MSNF)	ISO 1737 | IDF 13	Calculation from total solids content, fat content and sucrose content Gravimetry, drying at 102°C and Gravimetry (Röse - Gottlieb) and Polarimetry	IV
Blend of sweetened condensed skimmed milk and vegetable fat (for products sweetened with sucrose only)	Milk protein in MSNF[13]	ISO 1737 | IDF 13	Calculation from total solids content, fat content, sucrose content and protein content Gravimetry, drying at 102°C and Gravimetry (Röse-Gottlieb) and Polarimetry and Titrimetry (Kjeldahl)	IV
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat	Total fat	ISO 1737 | IDF 13	Gravimetry (Röse -Gottlieb)	I

Reduced fat blend of sweetened condensed skimmed milk and vegetable fat (for products sweetened with sucrose only)	Milk solids -not -fat[13] (MSNF)	ISO 1737 | IDF 13	Calculation from total solids content, fat content and sucrose content Gravimetry, drying at 102°C and Gravimetry (Röse -Gottlieb) and Polarimetry	IV
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat (for products sweetened with sucrose only)	Milk protein in MSNF[13]	ISO 1737 | IDF 13	Calculation from total solids content, fat content, sucrose content and protein content Gravimetry, drying at 102°C and Gravimetry (Röse - Gottlieb) and Polarimetry and Titrimetry (Kjeldahl)	IV
Cheese	Milkfat	ISO 1735 | IDF 5	Gravimetry (Schmid-Bondzynski - Ratzlaff)	I
Cheeses, individual	Milkfat in dry matter	ISO 1735 | IDF 5	Calculation from dry matter content and fat content Gravimetry, drying at 102°C and Gravimetry	I
Cheeses in brine	Milkfat in dry matter (FDM)	ISO 1735 | IDF 5	Calculation from dry matter content and fat content Gravimetry, drying at 102°C and Gravimetry (Schmid- BondzynskiRatzlaff)	I
Cottage cheese	Fat-free dry matter	ISO 1735 | IDF 5	Calculation from dry matter content and fat content Gravimetry, drying at 102°C Gravimetry (Schmid-Bondzynski-Ratzlaff)	I
Cottage cheese (for samples containing lactose up to 5%)	Milkfat in dry matter	ISO 1735 | IDF 5	Calculation from dry matter content and fat content Gravimetry, drying at 102°C and Gravimetry (Schmid-Bondzynski-Ratzlaff)	I

Cottage cheese (for samples containing lactose up to 5%)	Milkfat	ISO 1735 | IDF 5	Gravimetry (Schmid-Bondzynski-Ratzlaff)	I
Cream cheese	Moisture on fat-free basis	ISO 1735 | IDF 5	Calculation from fat content and moisture content Gravimetry drying at 102°C (forced air oven) Gravimetry (Schmid-Bondzynski-Ratzlaff)	I
Dairy permeate powders	Milkfat	ISO 1736 | IDF 9	Gravimetry (Röse-Gottlieb)	I
Edible casein products	Milkfat (Total fat)	ISO 5543 | IDF 127	Gravimetry (Schmid-Bondzynski-Ratzlaff)	I
Milk powders and cream powders	Milkfat	ISO 1736 | IDF 9	Gravimetry (Röse-Gottlieb)	I
Mozzarella	Milkfat in dry matter – with high moisture	ISO 1735 | IDF 5	Calculation from dry matter content and fat content Gravimetry, drying at 102°C and Gravimetry (Schmid-Bondzynski-Ratzlaff)	I
Mozzarella	Milkfat in dry matter – with low moisture	ISO 1735 | IDF 5	Calculation from dry matter content and fat content Gravimetry, drying at 102°C and Gravimetry (Schmid-Bondzynski-Ratzlaff)	I
Whey cheeses by coagulation	Milkfat	ISO 1735 | IDF 5	Gravimetry (Schmid-Bondzynski-Ratzlaff)	I
Whey cheeses by coagulation	Milkfat in dry matter	ISO 1735 | IDF 5	Calculation from fat content and dry matter content Gravimetry (Schmid-Bondzynski-Ratzlaff) Gravimetry, drying at 102°C	I
Fermented milks	Milkfat	ISO 1211 | IDF 1	Gravimetry (Röse-Gottlieb)	I
Cream	Milkfat	ISO 2450 | IDF 16	Gravimetry (Röse-Gottlieb)	I

Creams lowered in milkfat content	Milkfat	ISO 2450 | IDF 16	Gravimetry (Röse-Gottlieb)	I
Evaporated milks	Milkfat	ISO 1737 | IDF 13	Gravimetry (Röse-Gottlieb)	I
Evaporated milks	Milk Protein in MSNF[13]	ISO 1737 | IDF 13	Calculation from total solids content, fat content and protein content Gravimetry, drying at 102°C and Gravimetry (Röse-Gottlieb) and Titrimetry (Kjeldahl)	I
Sweetened condensed milk	Milkfat	ISO 1737 | IDF 13	Gravimetry (Röse-Gottlieb)	I
Sweetened condensed milks (for products sweetened with sucrose only)	Milk Protein in MNSF[13]	ISO 1737 | IDF 13	Calculation from total solids content, fat content, sucrose, and protein content Gravimetry, drying at 102°C and Polarimetry Gravimetry (Röse-Gottlieb) Titrimetry (Kjeldahl)	I
Whey cheeses by concentration (carbohydrate contents below 5%)	Milkfat (Total fat)	ISO 1854 | IDF 59	Gravimetry (Röse Gottlieb)	I
Whey cheeses by concentration (for carbohydrate content under 5%)	Milkfat in dry matter (total fat in dry matter)	ISO 1854 | IDF 59	Calculation from fat content and dry matter content Gravimetry (Röse Gottlieb) Gravimetry, drying at 88°C	I
Whey powders	Milkfat	ISO 1736 | IDF 9	Gravimetry (Röse-Gottlieb)	I
Infant formula	Total fat	ISO 8381|IDF 123	Gravimetry (Röse-Gottlieb)	I

Part 3

Cereals, pulses, and legumes workable package for consideration by EWG

Commodity	Provision	Codex Standard	Method	Principle	Type	Committee	*Comments*
Pearl millet flour	Colour	CXS 170-1989	*Modern Cereal Chemistry*, 6th Ed., D.W. Kent-Jones and A.J. Amos (Ed.), pp. 605-612, Food Trade Press Ltd, London, 1969.	Colorimetry ~~using~~ (specific colour grader)	IV	CCCPL	Colour-grading equipment used in method is no longer available, reconsideration of provision/method suggested by reviewers.
Quinoa	Moisture ~~content~~	CXS 333-2019	ISO 712 ~~/~~ AACCI 44-15.02	Gravimetry **(oven drying at 130 – 133°C)**	I	CCCPL	Methods are not identical, both methods endorsed by CCMAS as identical in a previous meeting. Further consideration may be needed, given the larger sample size with AACCI 44-15.02

Commodity	Provision	Codex Standard	Method	Principle	Type	Committee	*Comments*
Quinoa	Protein ~~(N x 6.25 in dry weight basis~~)	CXS 333-2019	ISO 20483 ~~ISO 1871~~ and ISO 712	Calculation from moisture and Titrimetry (Kjeldahl digestion)	~~IV~~ I	CCCPL	Validation information for ISO 1871 required, data ae available and anticipated to be shared for review. While ISO 20483 is acceptable for cereals, quinoa is a pseudocereal and it is not considered as a good fit.
Sorghum flour	Ash	CXS 173-1989	AOAC 923.03 / ISO 2171 ICC 104/1 and ISO 712 / ICC 110/1	Calculation from moisture and Gravimetry (incineration ~~at 550°C~~)	~~I~~	CCCPL	
Sorghum flour	Colour	CXS 173-1989	*Modern Cereal Chemistry*, 6th Ed., D.W. Kent-Jones and A.J. Amos (Ed.), pp. 605-612, Food Trade Press Ltd, London, 1969.	Colorimetry ~~using~~ (specific colour grader)	IV	CCCPL	Colour-grading equipment used in method is no longer available, reconsideration of provision/method suggested by reviewers.
Sorghum grains	Ash	CXS 172-1989	AOAC 923.03 / ISO 2171 ICC 104/1 and ISO 6540	Calculation from moisture and Gravimetry (incineration ~~at 550°C~~)	I	CCCPL	

Commodity	Provision	Codex Standard	Method	Principle	Type	Committee	*Comments*
Soy protein products	Fat	CXS 175-1989	CAC/RM 55 - Method 1	Gravimetry (extraction)	I	CCVP	Method is not available Replacement requested, none identified to date
Soy protein products	Protein; excluding added vitamins, minerals, amino acids, and food additives ~~(N x 6.25)~~	CXS 175-1989	AOAC 955.04D	Titrimetry (Kjeldahl digestion)	I	CCVP	Recommend revoke method and replace – mercury used Replacement requested, none identified to date
Vegetable protein products	Fat	CXS 174-1989	CAC/RM 55 - Method 1	Gravimetry (extraction)	I	CCVP	Method is not available Replacement requested, none identified to date
Vegetable protein products	Crude Protein; excluding added vitamins, minerals, amino acids, and food additives	CXS 174-1989	AOAC 955.04D	Titrimetry (Kjeldahl digestion)	~~II~~ I	CCVP	Recommend revoke method and replace – mercury used Replacement requested, none identified to date

Commodity	Provision	Codex Standard	Method	Principle	Type	Committee	*Comments*
Gari	Particle size (classification)	CXS 151-1989	ISO 2591-1	Sieving	I	CCCPL	Recommended for removal, however, classification determined by sieve size used. ISO 2591 provides general guidance on sieving protocols, but is not specific to CPL.
Edible Cassava flour	Particle size	CXS 176-1989	ISO 2591-1	Sieving	I	CCCPL	Recommended for removal, however, classification determined by sieve size used. ISO 2591 provides general guidance on sieving protocols, but is not specific to CPL.
Degermed maize (corn) meal and maize (corn) grits	Ash	CXS 155-1985	AOAC 923.03 **/** ISO 2171 ICC 104/1 and ICC 110/1	Calculation from moisture and Gravimetry **(incineration ~~at 550°C~~)**	I	CCCPL	
Wheat flour	Ash	AOAC 923.03 **/** ISO 2171 ICC 104/1 and ISO 712 / ICC 110/1	Gravimetry (incineration at 550°C)	I			

Commodity	Provision	Codex Standard	Method	Principle	Type	Committee	*Comments*
Whole maize (corn) meal	Ash	CXS 154-1985	AOAC 923.03 **/** ISO 2171 ICC 104/1 and ICC 110/1	Calculation from moisture and Gravimetry **(incineration ~~at 550°C~~)**	I	CCCPL	

APPENDIX III

INFORMATION DOCUMENT

PROCEDURES FOR THE ESTIMATION OF MEASUREMENT UNCERTAINTY

(for publication on the Codex website)

1 Introduction

A measurement result should be accompanied by information regarding its uncertainty. Such information provides an indication of the quality of the measurement result and allows meaningful comparison to other measurement results or reference values. Without a statement of measurement uncertainty, a measurement result is essentially incomplete and cannot be properly interpreted.

This document provides information regarding those sources of uncertainty which originate in the laboratory itself, i.e. in connection with the procedures and conditions starting with the laboratory sample and ending with the measurement result. In particular: the question of sampling uncertainty and the extent to which laboratory samples are representative of the content in the container will not be addressed in this information document. Such questions are addressed in the *General Guidelines on Sampling* (CXG 50-2004) [13].

Measurement uncertainty is defined as a parameter "…that characterizes the dispersion of the values that could reasonably be attributed to the measurand", see 2.2.3 in GUM [1]. This document aims to clarify what is meant by this definition and to provide the information which is necessary to understand how different approaches for the evaluation of measurement uncertainty relate to one another. This should allow the reader to make informed decisions regarding the best procedure to adopt in any given case.

Accordingly, the present document provides background information and clarifies basic notions which are central to a correct evaluation and interpretation of measurement uncertainty. First, the top-down and bottom-up approaches are described and compared. Then, the basic model for the top-down approach is presented. This constitutes a convenient framework within which to elucidate some of the basic conceptual aspects of measurement uncertainty. In the course of the discussion, the term *measurand* will be explained and the relationship between the top-down and bottom-up approaches will be further clarified on the basis of a more general classification of uncertainty sources. The question of the statistical uncertainty in estimating dispersion parameters – such as standard deviation values – will be addressed; and the effect of the number of observations on this statistical uncertainty will be examined. Specific designs for the evaluation of the different components of the top-down approach will then be provided, including designs for the evaluation of subsampling and matrix effects. Finally, examples will illustrate how measurement uncertainty influences sampling plans.

2 Top-down versus bottom-up approaches

The term "bottom-up approach" is used to denote any approach in which the measurement uncertainty is calculated on the basis of an equation expressing the relationship between input variables and the measurement result. In the phrasing from Section 4.1.1 of the *Guide to the expression of uncertainty in measurement* (GUM) [1]: In most cases, a measurand Y is not measured directly, but is determined from N other quantities $X_1, X_2, \dots, X_N$ through a functional relationship (model) f:

$$Y = f(X_1, X_2, \dots, X_N)$$

It must be emphasized that, in this approach, the measurement result Y is *calculated* from the input variables $X_1, X_2, \dots, X_N$. Analyte concentration is an example of a measurement result; optical density, peak area and signal height are examples of input variables.

An alternative approach – described e.g. in EURACHEM/CITAC Guide CG4 [2] and in ISO 21748 [4] – consists in making use of available *method validation* data. In the words of Section 7.6.1 in the EURACHEM Guide [2]: "A collaborative study carried out to validate a published method […] is a valuable source of data to support an uncertainty estimate." In this approach, there is no "functional relationship" between input variables and the measurement result. Rather, results are obtained under different measurement conditions, and total observed variation is partitioned into individual components. This approach is often referred to as the *top-down* approach.

In order to obtain measures of precision which can subsequently be used to "support an uncertainty estimate" following the top-down approach, two main types of experiments can be conducted: single-lab (in-house) and multi-lab (collaborative) studies. It must be emphasized that precision measures obtained in these two types of studies are not always comparable. Nonetheless, if relevant uncertainty sources have not been taken into account, it is often expedient to complement the information from a multi-lab study by subsequent single-lab experiments.

The main distinction between the two approaches is that whereas the bottom-up approach starts from a physico-chemical consideration of the actual measurement mechanism, the top-down approach starts from a data set in which the variation between different measurement results is directly observable. In this sense, it can be said that the bottom-up approach is *theoretical* whereas the top-down approach is *empirical*.

A related distinction is that, in the bottom-up approach, the starting point is the relationship between the measurement result and input variables, whereas, in the top-down approach, the starting point is the relationship between total variation and individual components of variation.

Finally, another distinction between both approaches is that while the number of components in the top-down approach is usually low[1], the number of input variables in the bottom-up approach can be quite high. For this reason, in the bottom-up approach, it will often be impractical to conduct an experiment in which estimates for the uncertainties associated with all the input variables can be reliably obtained. Indeed, the bottom-up approach explicitly allows the inclusion of *prior information* regarding the size of the errors which can be expected to arise in connection with each source (Type B evaluation).

In the case of the bottom-up approach, there are two options for the calculation of the combined (i.e. total) measurement uncertainty: performing a linear approximation and the Monte Carlo Method (MCM).

Bottom-up approach: linear approximation

The first option is often referred to as the law of propagation of uncertainty. In the case that there are no correlations between the different input variables, the combined measurement uncertainty – expressed as a standard deviation – is obtained as follows:

$$u_c = \sqrt{\sum_{i=1}^{N} c_i \cdot u_i^2}$$

where u_c denotes the combined uncertainty, u_i denotes the uncertainty associated with input variable i and c_i denotes the corresponding sensitivity coefficient, usually obtained via partial differentiation, i.e. $c_i = \left(\frac{\partial f}{\partial X_i}\right)^2$, see 5.1.2 and 5.1.3 in GUM [1].

Bottom-up approach: Monte Carlo method

The second option consists in applying a Monte Carlo method (MCM). This method is preferable to linear approximation and can be used to verify measurement uncertainty estimates obtained via linear approximation. The Monte Carlo method can briefly described as a computer simulation of the measurement process or (in statistical terms) as "repeated sampling from the probability density functions of the X_i and the evaluation of the model in each case," see 5.9.1 in [3]. This option is also referred to as the propagation of distributions. In practice, the implementation of this option requires software, since the number of simulation runs (i.e. the number of times each input variable is sampled) is typically on the order of 10^6. If the function f is highly nonlinear, the used of MCM is recommended. For instance, in the case of standard addition, the model is

$$Y = \frac{a}{b}.$$

In this model, b denotes the slope parameter, calculated as

$$b = \frac{\sum_{i=1}^{n}(x_i - \bar{x}) \cdot (y_i - \bar{y})}{\sum_{i=1}^{n}(x_i - \bar{x})^2}$$

where the x_i denote the added standard concentrations (with mean value $\bar{x}$) and the y_i denote the corresponding response values (with mean value $\bar{y}$); and a denotes the intercept, calculated as

$$a = \bar{y} - b \cdot \bar{x}.$$

The uncertainty values of the individual x_i variables are taken from the certificates of the reference standard substances of materials, while the uncertainty values for the y_i variables are obtained from the regression analysis[2] (residual standard deviation).

For such a model, the results obtained via linear approximation and via MCM can differ considerably. The MCM calculation will also show whether the distribution of the measurand is asymmetric. For instance, in the case of standard addition, the distribution for the measurand $\mathrm{Y} = \frac{\mathrm{a}}{\mathrm{b}}$ is typically right-skewed:

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1 The number of components follows directly from the experimental design of the method validation study.

2 Strictly speaking, a linear regression approach that takes into account errors in observations in both the x- and y-axes (e.g. Deming regression) should be applied.

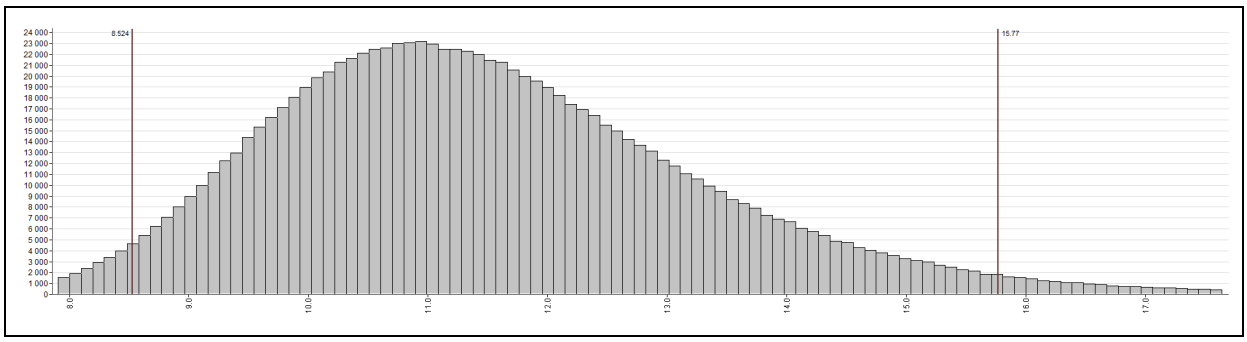

Figure 1: Right-skewed distribution for the standard addition measurand $Y = \frac{a}{b}$ obtained via 10^6 MCM simulation runs.

If it cannot be ensured that all relevant uncertainty contributions are included in the Monte Carlo model, it is suggested to resort to the top-down approach, as described below.

Top-down approach

In the case of the top-down approach, the total measurement uncertainty is obtained by summing different variance components, such as between-laboratory variance and repeatability variance. The number of replicate measurements should be taken into consideration. For instance, in the simplest case, the total standard uncertainty is obtained as

$$u = \sqrt{s_L^2 + \frac{s_r^2}{n_r}}$$

where s_L denotes the between-laboratory standard deviation, s_r denotes the repeatability standard deviation and n_r denotes the number of replicates whose mean value is taken as the final measurement result. For further information, the reader is referred to ISO 21748 [4].

3 Basic model for the top-down approach

In this section, the basic model for the top-down approach is discussed. The model is premised on the assumption that data from an interlaboratory validation study (also known as a collaborative study) are available. Such a study is conducted in order to characterize the performance of an analytical method. In particular, the characterization of the *precision*[3] of an analytical method can be used "to support an uncertainty estimate". The reader is referred to the ISO 5725 series – in particular to Part 2 [5] – for background information.

The basic model is as follows:

$$\begin{aligned}\text{Measurement value } Y \\ = \ & true\ value\ +\ method\ bias\ (average\ across\ labs\ and\ matrices) + \ matrix\text{-}specific\ bias \\ + \ & laboratory\ bias + \ repeatability\ error\end{aligned}$$

For further details, the reader is referred to [6] and [7]. In the case of an in-house method, the laboratory bias term is replaced by a term representing intermediate effects (whether via measurements performed on different days, or via a factorial design), see ISO TS 23471 [20] and Section 9.1 below.

In the following, the individual terms of the basic model are discussed.

True value

In general, the true value is not known. It can be estimated by averaging e.g. across methods, samples and laboratories (this will often result in a mere approximation). However, it is crucial to note that in the GUM [1], measurement uncertainty is defined *without any reference to a true value*; rather, it is defined as a parameter "… that characterizes the dispersion of the values which could reasonably be attributed to the measurand", see 2.2.3 in GUM [1]. This definition has since been adopted in all other relevant standards and guidance documents (EURACHEM [2], VIM [8]). This does not mean that the true value no longer plays a role in the evaluation of measurement uncertainty.

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3 Precision is defined (paraphrasing 2.15 in [8]) as the degree of agreement between independent measurement results obtained under specified conditions. For instance, reproducibility precision characterizes the agreement between results from different laboratories, while repeatability precision characterizes the agreement between results obtained under near-identical conditions in the same laboratory. Precision can be used to derive a measurement uncertainty estimate – but it must not be confused with measurement uncertainty.

However, it is not the (unavailable) difference between true value and measurement result, but *the uncertainty of bias correction* which must be taken into account in the evaluation of measurement uncertainty. In other words, the focus shifts from the (unavailable) true value itself to the uncertainty in the estimation of the bias. Note that if a certified reference value is available along with a reference uncertainty value, the latter can be included in the uncertainty of bias correction.

Method bias (average across labs and matrices)

The method bias can be estimated by averaging across laboratories and matrices. A prerequisite is that, within each participating laboratory, measurements are performed under appropriate conditions, with appropriate equipment and trained personnel. Any available information from (certified) reference materials should be taken into consideration. As explained in the discussion of the true value, the corresponding contribution to the calculation of measurement uncertainty will consist in the uncertainty in the estimate of this bias.

Matrix-specific bias (matrix mismatch)

In many cases, a method's bias depends on the matrix being examined. In other words: bias varies from one matrix to another. Such effects occur when the extraction of analyte is affected by the matrix, so that a part of the analyte is not recovered; or when a part of the matrix is extracted along with the analyte and interacts with the measurement's physico-chemical mechanism, resulting in a bias. The corresponding component of total variability is referred to as the matrixmismatch component. It is important to note that all the uncertainty sources listed in Section 7 contribute to this term of the basic model.

Laboratory bias

In many cases, a method's bias depends on the laboratory which is performing the measurement. In other words, the bias varies from laboratory to laboratory. It is often impractical or insufficient to estimate laboratory bias and perform a correction of laboratory bias[4]. Accordingly, in order to take laboratory bias into consideration in estimating measurement uncertainty, it is often expedient to resort to the expected range of laboratory bias, characterized via the laboratory standard deviation (one of the components of the reproducibility precision). A prerequisite is that, within each laboratory that participated in the validation study, measurements were performed under appropriate conditions, with appropriate equipment and trained personnel.

Repeatability error

This term represents variation across replicate measurements (i.e. independent measurements performed under near-identical test conditions).

Note regarding the case that the precision depends on the concentration level

When there is a known relationship between precision (e.g. in-house reproducibility) and concentration, it is possible to apply an approach based on a clear distinction between, on the one hand, random variation between test results at a given concentration level, and, on the other, the range of values which can "reasonably be attributed to the measurand," i.e. the measurement uncertainty. This approach gives rise quite naturally to asymmetrical measurement uncertainty intervals in cases of relatively poor precision (say, greater than 10 %) and heteroscedasticity (e.g. constant *relative* in-house reproducibility). This approach is also described in Annex E of ISO TS 23471 [20].

Note regarding bias and (certified) reference materials

Bias can be taken into account in two different ways in measurement uncertainty.

Case 1: Uncertainty of bias correction

If (certified) reference material is available, then an estimate of bias can be obtained, and a bias correction can be performed. If a bias correction is performed, the uncertainty of the bias correction should be included in the measurement uncertainty. In the simplest case, the uncertainty of the bias correction has two components: the uncertainty of the mean value $\bar{y}$ obtained in the experiment, and the uncertainty of the reference value x. (In this simplest case, the estimate of bias is $\bar{y} - x$). The uncertainty of the reference value x may be a value available from a certificate (e.g. in the case of certified reference material); however, it may also be derived from precision data from a validation study.

Case 2: Prediction range of bias

If several estimates of bias are available, then the expected range of bias can be included in the estimate of measurement uncertainty. This is typically done in the top-down approach, when the measurement uncertainty estimate is derived from the precision data of a collaborative study.

[4] This is principally due to the absence of appropriate reference material, intermediate precision effects on bias estimates and the fact that laboratory bias may vary from one matrix to the next.

Indeed, for each sample, the difference between each laboratory mean and the overall mean can be considered an estimate of laboratory bias, and the between-laboratory standard deviation s_L then characterizes the expected range of laboratory bias. Similarly, if data obtained on the basis of several matrices are available, the variation of laboratory bias across matrices can be characterized. In this approach, the availability of (certified) reference material is not necessary; nor is the performance of bias correction required. (Though the interpretation of the variation of bias will depend on whether correction was previously performed and on whether the variation was calculated in relation to a reference value.)

4 Specifying the measurand

The concept "measurand" clearly plays a central role in the definition of measurement uncertainty and will shed further light on the connection between validation data and measurement uncertainty.

Leaving aside the technicalities of the definition of a measurand[5], it is sufficient to note that the specification of a measurand has three separate components:

- specification of a property, e.g. *mean arsenic concentration.* Note that the concept "analyte" corresponds to this part of the specification of the measurand
- specification of a phenomenon, body or substance which the property is associated with, e.g. *a given batch of apple juice*. Note that the concept "matrix", used in the previous section, corresponds to this part of the specification of the measurand
- and specification of a reference framework regarding the manner in which the property is characterized, e.g. [ng/ml]

Loosely phrased, specifying a measurand thus involves stating (1) *what* is to be measured, (2) what is it to be measured *in*, and (3) *how* should the measurement result be expressed in order to ensure comparability to other measurement results or relevant values?

In particular, the specification of the measurand should include information as to whether analyte concentration is to be measured in a laboratory sample or in a "larger sample" or a batch of products in a container. Only in the latter case is *sampling* uncertainty relevant (see Section 7 for an overview of the different sources of uncertainty). Similarly, if measurement results from several laboratory samples are used to assess the conformity of bulk material from a container, it is the measurement uncertainty of the mean value across the results corresponding to the individual laboratory samples which is relevant.

More generally, while measurement uncertainty is always determined on the basis of the laboratory sample, it is nevertheless important to include all available information about the laboratory sample in the evaluation of measurement uncertainty, e.g.

- Where does the material come from (e.g. container)?
- Have other samples from the same origin been tested?
- What is the intended use of the measurement result (e.g. conformity assessment for the individual laboratory sample or for the container)?

For example, determining the contribution to uncertainty which arises from the material's heterogeneity (e.g. fundamental variability, see Section 9.4) may require a considerable amount of work, depending on the analyte, concentration and grain/particle size. If the origin of the material is known, it may be possible to use previously obtained results regarding the heterogeneity contribution to uncertainty instead of obtaining a new estimate from scratch.

The specification of the measurand should also make it possible to determine whether bias/recovery correction is required, and what form this correction should take. For example, if the measurand is specified in terms of the amount of analyte recovered, then recovery correction may not be appropriate. On the other hand, if the measurand is specified in terms of the total amount of analyte present in a test sample, then recovery correction may be necessary.

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[5] In the VIM [8], measurand is defined (definition 2.3) as "quantity intended to be measured". Quantity, in turn, is defined (definition 1.1) as "property of a phenomenon, body, substance, where the property has a magnitude that can be expressed as a number and a reference". An example given directly under this definition is "amount-of-substance concentration of ethanol in wine sample i". The term "reference" in this definition is explained in NOTE 2 as: "A reference can be a measurement unit, a measurement procedure, a reference material, or a combination of such."

Finally, it may be impractical or impossible to provide an exhaustive specification of the measurand. For this reason, it may be necessary to include an extra component of measurement uncertainty, called “definitional uncertainty” (see definition 2.27 in VIM [8]), in order to account for any ambiguity (“finite amount of detail”) in the specification of the measurand. However, in most cases, the definitional uncertainty can be considered negligible.

5 Relation between measurand and validation data

If the results of a validation study are to be used to determine measurement uncertainty, it must be ensured that the study refers to the same measurand.

Example 1: Measurement uncertainty is being evaluated in a given laboratory for a measurand specified in terms of analyte concentration in test samples. The analytical method used has been validated for the same analyte, but on the basis of extracts rather than test samples. In other words, the measurand for the validation study is analyte concentration in extracts. It follows that the measurand for which measurement uncertainty must be evaluated is different from the measurand from the validation study. Accordingly, the measurement uncertainty cannot be evaluated on the basis of the characterization of the dispersion of measurement results from the validation study.

Example 2: Measurement uncertainty is being evaluated in a given laboratory for a measurand which is specified in terms of a range of matrices. The analytical method used has been validated for the same analyte, but for only one of the matrices. It follows that the measurand for which measurement uncertainty must be evaluated is different from the measurand from the validation study. Accordingly, the measurement uncertainty cannot be evaluated on the basis of the characterization of the dispersion of measurement results from the validation study (the matrix mismatch term is missing, see Section 9.2).

The conditions under which validation data can be used to support a measurement uncertainty estimate can be stated as follows:

If…

the measurement result is obtained using a validated method

and the *measurand* is included in the scope of the validation

and precision (in particular, in-house reproducibility standard deviation) within the laboratory which is evaluating measurement uncertainty is comparable to the method’s precision as characterized in the validation study

then…

→ the precision estimates from the validation study can be used in the calculation of measurement uncertainty.

Note: These precision estimates can also be used to derive an estimate of the uncertainty of method bias correction.

In order to check and provide evidence of competence in the application of the method and to ensure adequate precision in the laboratory which is evaluating measurement uncertainty, it may be necessary to perform a verification study.

The reader is referred to Section 7 in EURACHEM [2] for further guidance regarding using validation data in the evaluation of measurement uncertainty.

6 Empirical versus rational methods

In the definition of the measurand, the specification of the property must include sufficient information to allow an appropriate reference (see 1.1 in the VIM [8]) to be selected. In particular, it is important to distinguish between

- Empirical method (type I methods in the Codex system)
- Rational method (type II-IV methods in the Codex system)

In Section 5.4 of EURACHEM [2], the following explanation is provided: *"In analytical measurement, it is particularly important to distinguish between measurements intended to produce results which are independent of the method used, and those which are not so intended. The latter are often referred to as empirical methods or operationally defined methods."*

In Section 5.5 of the same document, it is explained that non-empirical methods are sometimes called rational methods. This distinction is closely related to that between *operationally defined* and *non-operationally defined* measurands found in Section 9.2.3 of ISO Guide 35 [9]. The reader is also referred to Section 3.1 in the EURACHEM Guide to Metrological Traceability in Chemical Measurement [21].

As far as the evaluation of measurement uncertainty is concerned, this distinction has the following important implication: for *empirical* methods (*operationally defined* measurands), there is no method bias term in the basic model for the top-down approach described in Section 3. (Please note that the bottom-up approach does not allow the distinction *method* versus *other* bias components).

7 Uncertainty sources in the top-down and bottom-up approaches

In the *top-down* approach, total variation observed in a data set is partitioned into different components. In the *bottom-up* approach, the total uncertainty is obtained from uncertainty values associated with individual input variables. The following question arises: what is the *relationship* between the components from a top-down model and the uncertainty sources included in a bottom-up model?

In order to answer this question, an overview of different types of uncertainty sources – *independently of the approach* – is now provided. The intention is to distinguish broad categories of uncertainty sources. Apart from shedding further light on the relationship between the top-down and bottom-up approaches, this overview may prove useful for determining which sources may be relevant in any given case, and whether all relevant sources have been included in the evaluation of measurement uncertainty.

Sources of uncertainty are conveniently classified under six main headings:

- Sampling (The question of sampling uncertainty is not addressed in the present document. The reader is referred to CXG 50-2004 [13])
- Storage/transportation
- Subsampling
- Measurement conditions
- Measurement procedure
- Computational effects

Source of uncertainty	Role in measurement uncertainty
Sampling	If the measurand is defined in terms of e.g. analyte concentration in a container or in a batch of products, then sampling is required, and its contribution to measurement uncertainty must be assessed, see Section 7.6 in ISO 17025 [10]. If the measurand is defined in terms of a single test material (laboratory sample), then there is no contribution to uncertainty due to sampling. There may be a contribution from subsampling, however (i.e. obtaining test portions from the laboratory sample). *Fundamental variability* is one of the "subcomponents" of sampling uncertainty, see the discussion in Section 9.4.
Storage/transportation	If different storage or shipping conditions have an effect on measurement results, then the corresponding contribution to the total uncertainty must be taken into account.
Subsampling	This term denotes taking test portions from the laboratory sample. If the latter is not homogeneous (finely ground in case of solid matter, mixed or agitated in case of liquids and semi-solids), then it cannot be ensured that the subsampling uncertainty is negligible. Accordingly, appropriate homogenisation is required before subsampling in order to reduce this uncertainty source. *Fundamental variability* is one of the "subcomponents" of subsampling uncertainty, see the discussion in Section 9.4.
Measurement conditions	It must be emphasized that the term measurement as used here includes any sample preparation and clean-up procedures. If different measurement conditions (e.g. different time of year, different technician, different reagents, different equipment) contribute to measurement uncertainty, this source must be taken into consideration.
Measurement procedure	This term denotes the intrinsic or irreducible uncertainty component associated with the physical/chemical/biochemical mechanisms involved in the measurement procedure (including sample preparation and clean-up procedures), e.g. extraction efficiency. The input variables in the bottom-up approach can be considered to belong under this heading.
Computational effects	Inaccurate calibration model and calculation methods, peak integration procedures and rounding will also contribute to measurement uncertainty.

Note regarding quantitative microbiological methods and estimating measurement uncertainty in accordance with ISO 19036

In ISO 19036 [24], a top-down approach is described. This approach distinguishes three components of measurement uncertainty: the technical uncertainty, the matrix uncertainty and the distributional uncertainty. The technical uncertainty represents sources of uncertainty described in Section 7 under the categories *Measurement conditions* and *Measurement procedure*. The technical uncertainty thus arises from the implementation of the method *per se*. The technical uncertainty does not include any variation arising from the heterogeneity of the laboratory sample. Such variation is represented by the matrix uncertainty. The matrix uncertainty as defined in ISO 19036 thus corresponds to the subsampling source of uncertainty listed in Section 7. Conceptually, it has no relation whatsoever to the matrix-specific bias term (matrix mismatch) described in Section 3. Finally, the distributional uncertainty arises from the fact that the number of discrete cells (colony forming units) may vary from test portion to test portion, even if the laboratory sample is perfectly homogenous. The distributional uncertainty is thus related to the fundamental variability discussed in Section 9.4. It should be mentioned that the following uncertainty components are not covered by ISO 19036:

- method or lab bias (there are no "true values" for microbiological measurands)
- sampling

8 Requirements regarding data size

If a standard deviation is calculated on the basis of a series of measurement results, how well does it characterize the actual dispersion of the values? Indeed, if several measurement series are performed and a separate standard deviation value is calculated for each, these standard deviation values will differ. In other words, a given standard deviation, obtained on the basis of empirical data, only represents an *estimate* of the "true" standard deviation.[6]

The confidence interval for a standard deviation can be obtained by means of the following Excel formula: SQRT((N-1)/CHISQ.INV(p,N − 1)), where p is the probability value (e.g. 0.025 or 0.975) and N is the number of laboratories or the number of tests inside the single laboratory. This Excel formula corresponds to the following mathematical formulas for the lower and upper limits (LCL and UCL) of a 95 % confidence interval given a standard deviation estimate s: LCL = $\sqrt{\frac{\mathrm{N}-1}{\chi^2_{(\mathrm{N}-1,0.975)}}} \cdot s$ and UCL = $\sqrt{\frac{\mathrm{N}-1}{\chi^2_{(\mathrm{N}-1,0.025)}}} \cdot s$, where $\chi^2_{(\nu,\mathrm{p})}$ denotes the p-quantile of a chi-squared distribution with ν degrees of freedom.

It is recommended that standard deviations be computed on the basis of a minimum of $N = 12$ values (corresponding to $\nu = 11$ degrees of freedom for the estimation of the standard deviation), in which case $\chi^2_{(\mathrm{N}-1,0.975)} = \chi^2_{(11,0.975)} = 21.92$ and $\chi^2_{(\mathrm{N}-1,0.025)} = \chi^2_{(11,0.025)} = 3.82$, and the confidence interval for the standard deviation is $[0.71 \cdot s, 1.70 \cdot s]$.

As far as the simultaneous estimation of e.g. between-laboratory (or between-matrix) standard deviation and repeatability standard deviation is concerned, this recommendation means that measurement results from at least 12 laboratories (or matrices) should be available, each with at least two replicates per laboratory (or matrix).

It is required that data from at least 8 laboratories must be available (see Section 6.3.4 in ISO 5725-1 [18] where 8-15 laboratories is proposed as a "common" figure).

In the case that different uncertainty sources are *simultaneously* taken into consideration, say in the bottom-up approach, the requirement regarding data size can be applied via the Welch-Satterthwaite formula, see [1] Annex G, G.4.1. More specifically: take the case that 2 different uncertainty sources are included in the calculation of the combined uncertainty, u_1 and u_2. Say that each was obtained by applying the formula for the sample standard deviation on the basis of n_1 and n_2 measurement results, respectively. The number of degrees of freedom for the combined uncertainty can then be computed as

$$\text{Degrees of freedom for combined uncertainty} = \frac{(u_1^2/n_1 + u_2^2/n_2)^2}{\frac{(u_1^2/n_1)^2}{n_1 - 1} + \frac{(u_2^2/n_2)^2}{n_2 - 1}}$$

The recommendation is to ensure a minimum of 11 degrees of freedom for the combined uncertainty.

In the case that prior information is used for an individual u_i value (Type B variable) and that no information regarding data size is available, it is suggested to use $n_i = 7$; the uncertainty which corresponds to this data size is intended to reflect the fact that, in the case of Type B variables, distributional assumptions are often based on "educated guesses."

Example of the application of the Welch-Satterthwaite formula

Take the case that measurement uncertainty must be evaluated on the basis of the following functional relationship, where the measurement result Y is expressed as a function of 4 input variables:

$$Y = f(X_1, X_2, X_3, X_4) = X_1 + X_2 + X_3 + X_4$$

Table 1: Data size and uncertainty values for the input variables

Input variable	Type	n	u^2
X_1	A	3	4
X_2	B	30	15
X_3	B	30	15
X_4	B	Not available Take $n_4 = 7$	5

[6] Table 3 in the *Guidelines on Estimation of Uncertainty of Results* (CXG 59-2006) [11] provides expected ranges for standard deviation estimates calculated from empirical data for different values of *N* (number of observations). Please note that expected ranges must not be confused with the confidence intervals.

The Welch-Satterthwaite formula can now be applied.

$$\text{Degrees of freedom for combined uncertainty} = \frac{(u_1^2/n_1 + u_2^2/n_2 + u_3^2/n_3 + u_4^2/n_4)^2}{\frac{(u_1^2/n_1)^2}{n_1 - 1} + \frac{(u_2^2/n_2)^2}{n_2 - 1} + \frac{(u_3^2/n_3)^2}{n_3 - 1} + \frac{(u_4^2/n_4)^2}{n_4 - 1}} = 9.4$$

We would suggest users of the Welch-Satterthwaite formula refer to the GUM [1] Annex G for a discussion of the formula and the range within which the calculated degrees of freedom should lie, since incorrect implementations commonly fall outside of this range.

9 Simple procedures for evaluating uncertainty components

If validation data are incomplete (i.e. some of the relevant sources of uncertainty have not been characterized), further experiments must be conducted before the top-down approach can be applied.

For instance, in a collaborative study, each participating laboratory should ideally receive samples representing different matrices and different analyte concentrations. However, due to restrictions in material availability, collaborative studies are often conducted on the basis of a single sample per participant. In such a case, almost no conclusions can be drawn regarding the impact of matrix effects. Accordingly, the characterization of the matrix-specific bias term from the basic model must often be performed in a separate experiment.

In the following, simple procedures are described for characterizing different components of variation – such as the matrix-specific bias.

More sophisticated procedures for simultaneously estimating several components of variation are provided in [12]. The reader is also referred to ISO TS 23471 [20], in which study designs are described for the evaluation of data obtained from several concentration levels in one laboratory; and to ISO 5725-3 [19], in which, mainly, alternative study designs are described for the evaluation of data from one concentration level in several laboratories.

9.1 PROCEDURE FOR CHARACTERIZING IN-HOUSE VARIATION

If the analytical method is an in-house method, then an in-house (single-lab) validation study is conducted. If validation data are incomplete or unavailable, in-house components of variation can be characterized on the basis of a further experiment (or QC data, as long as such data are available and have an appropriate structure).

Total in-house variation is called intermediate precision and should reflect all relevant uncertainty sources except matrix mismatch[7] – in particular, variation arising from different measurement conditions (i.e. operator, reagent batch, etc.) within the laboratory, along with repeatability.

The structure of the experimental or QC data must allow the distinction between in-house repeatability conditions and intermediate conditions (different day, different technician, different reagent batch, etc.). The uncertainty can then be calculated as follows:

$$u = \sqrt{s_I^2 - s_{r,inhouse}^2 + \frac{s_{r,inhouse}^2}{k}}$$

where s_I denotes the intermediate standard deviation, $s_{r,inhouse}$ denotes the repeatability estimate and k denotes the number of replicates whose mean value is taken as the final measurement result.

As explained in Section 8, it is recommended that, at a minimum, $N = 12$ different in-house measurement conditions (e.g. different days) be represented in the data set.

In the following example, we take the case that QC data are available for 20 different days. (If appropriate QC data are not available and a further experiment is required, $N = 12$ days are sufficient).

[7] By definition, intermediate precision does not include the variation of bias across matrices (matrix mismatch), see 2.22 in VIM [8]. If matrix mismatch is included, then the term in-house reproducibility is used.

Table 2: In-house QC data for the calculation of intermediate (in-house) and repeatability standard deviation values

	Result 1	Result 2
Day 1	10.72	12.29
Day 2	4.56	0.90
Day 3	8.79	9.75
Day 4	10.08	6.51
Day 5	12.29	11.32
Day 6	7.95	6.79
Day 7	13.06	14.54
Day 8	11.23	12.09
Day 9	7.31	9.51
Day 10	5.85	5.08
Day 11	7.48	9.12
Day 12	12.59	10.65
Day 13	7.55	6.59
Day 14	12.05	11.15
Day 15	4.86	6.48
Day 16	6.99	7.10
Day 17	7.40	6.75
Day 18	8.85	11.15
Day 19	11.93	10.17
Day 20	8.50	8.29

The between-day and repeatability standard deviation values are calculated as follows.

First we introduce the following notation: the days are indexed $i = 1, \ldots, m$ (in this example, $m = 20$); the replicates within each day are indexed $j = 1, n$ (in this example, $n = 2$); and the individual measurement results are denoted x_{ij}.

First, compute the overall mean value $\bar{x}$, and the day-specific mean values $\bar{x}_i$. Then compute the between-day sum of squares[8]:

$$SSB = n \cdot \sum_{i=1}^{m} (\bar{x}_i - \bar{x})^2$$

and the within-day sum of squares:

$$SSW = \sum_{i=1}^{m} \sum_{j=1}^{n} \left(x_{ij} - \bar{x}_i\right)^2$$

The in-house repeatability standard deviation $s_{r,inhouse}$ is then obtained as

$$s_{r,inhouse} = \sqrt{\frac{SSW}{m \cdot (n-1)}}$$

8 The following are standard formulas for the one-way analysis of variance with random effects.

and the between-day standard deviation s_D is obtained as

$$s_D = \sqrt{\frac{1}{n}\left(\frac{SSB}{m-1} - s^2_{r,inhouse}\right)}.$$

(If the value under the square root sign is negative, then $s_D = 0$.)

Finally, the intermediate (in-house) standard deviation is calculated as:

$$s_I = \sqrt{s_D^2 + s^2_{r,inhouse}}.$$

For the data from Table 2, the calculation results are as follows:

Table 3: Calculation of SSB and SSW on the basis of in-house QC data

Overall mean value $\bar{x}$	Day-specific mean values $\bar{x}_i$	Differences $\bar{x}_i - \bar{x}$	SSB	Differences $x_{ij} - \bar{x}_i$	Differences $x_{ij} - \bar{x}_i$	SSW
8.91	11.51	2.60	283.05	-0.79	0.79	29.95
	2.73	-6.18		1.83	-1.83	
	9.27	0.36		-0.48	0.48	
	8.29	-0.61		1.79	-1.79	
	11.80	2.90		0.49	-0.49	
	7.37	-1.54		0.58	-0.58	
	13.80	4.90		-0.74	0.74	
	11.66	2.75		-0.43	0.43	
	8.41	-0.50		-1.10	1.10	
	5.46	-3.44		0.39	-0.39	
	8.30	-0.61		-0.82	0.82	
	11.62	2.72		0.97	-0.97	
	7.07	-1.83		0.48	-0.48	
	11.60	2.69		0.45	-0.45	
	5.67	-3.24		-0.81	0.81	
	7.05	-1.86		-0.06	0.06	
	7.08	-1.83		0.32	-0.32	
	10.00	1.09		-1.15	1.15	
	11.05	2.14		0.88	-0.88	
	8.40	-0.51		0.10	-0.10	

The following precision estimates are obtained:

Table 4: Precision estimates obtained from in-house QC data

$s_{r,inhouse}$	s_D	s_I
1.22	2.59	2.86

9.2 PROCEDURES FOR CHARACTERIZING VARIATION OF BIAS ACROSS MATRICES (MATRIX MISMATCH)

In this section, a procedure for estimating the variation of bias across matrices is described. Such an estimate is necessary when

- on the one hand, a number of different matrices/sample types are included in the scope of the method
- and on the other hand, only few matrices/sample types were included in the validation study

It is assumed that, for any given matrix, heterogeneity between laboratory samples is negligible, and that the measurand is specified in terms of a number of matrices, from which N matrices are selected[9]. Selection should be based on the method's intended use/scope. As explained in Section 8, it is recommended that, at a minimum, $N = 12$ matrices be included.

A simple approach for characterizing variation of bias across matrices consists in spiking the N matrices and obtaining duplicate measurement results in a single laboratory for each matrix. In this manner, variation of bias between the matrices (matrix mismatch) can be distinguished from variation within each matrix (repeatability error). In this procedure, the matrix is modelled as a random effect, and the result is a standard deviation characterizing variation across all the matrices included in the specification of the measurand.

Example

Table 5: Data from an experiment for the calculation of the matrix mismatch effects (variation of bias across matrices)

	MV1	MV2
Matrix 1	114.51	112.24
Matrix 2	120.25	111.59
Matrix 3	88.46	86.62
Matrix 4	118.93	102.35
Matrix 5	74.06	80.91
Matrix 6	117.50	102.69
Matrix 7	120.96	109.35
Matrix 8	96.05	92.92
Matrix 9	98.43	87.09
Matrix 10	107.99	117.42
Matrix 11	117.34	126.87
Matrix 12	76.56	109.79

Applying the same calculation procedure as in Section 9.1, the following precision estimates are obtained:

Table 6: Precision estimates for the calculation of matrix mismatch

s_r	s_{matrix}
9.53	12.24

9.3 PROCEDURES FOR CHARACTERIZING BETWEEN-LABORATORY VARIATION

Procedure 1: Conduct an interlaboratory validation study with a minimum of $N = 12$ laboratories and with duplicate measurement results within each laboratory. It is necessary to ensure that heterogeneity between laboratory samples is negligible. In this manner, variation between the laboratories (lab bias) can be distinguished from variation within the laboratories (repeatability error). In this procedure, the laboratory is modelled as a random effect, and the result is a standard deviation characterizing variation across laboratories.

Example

Table 7: Data from an experiment for the calculation of the lab bias

	MV1	MV2

[9] For instance, a number of different apple types, or a number of different cattle breeds.

Lab 1	0.981	1.238
Lab 2	0.182	0.601
Lab 3	1.107	0.994
Lab 4	1.471	1.532
Lab 5	1.169	0.674
Lab 6	0.491	1.271
Lab 7	1.717	0.970
Lab 8	0.931	1.171
Lab 9	1.017	1.248
Lab 10	0.909	0.723
Lab 11	0.812	1.312
Lab 12	1.375	1.719

Applying the same calculation procedure as in Section 9.1, the following precision estimates are obtained:

Table 8: Precision estimates for the calculation of lab bias

s_r	s_{lab}
0.30	0.23

Procedure 2: If PT data are available, and a sufficient number of participants (ideally, at least 12) have used the same method (ideally, with replicate measurements within each laboratory) – then these data can be used to characterize variation across laboratories. In order to ensure neutral data evaluation and avoid conflicts of interest, the data should come from PT schemes run by competent authorities.

9.4 PROCEDURES FOR CHARACTERIZING CONTRIBUTIONS TO UNCERTAINTY FROM SAMPLE PREPARATION STEPS AND FROM SUBSAMPLING WITHIN THE LABORATORY

Procedures for characterizing contributions from sample preparation steps and from the heterogeneity of the laboratory sample

In a collaborative study conducted in accordance with ISO 5725-2, the repeatability component may or may not reflect contributions from

- sample preparation (all steps performed starting from the laboratory sample in order to obtain the test portion)
- subsampling variation due to the heterogeneity of the laboratory sample

In particular, if the collaborative study was conducted on the basis of reference material which

- is homogenous
- is sent to the laboratories in the form of test portions requiring no further sample preparation steps

then the repeatability estimate will reflect neither of these two sources of uncertainty.

For this reason, if either of these sources of uncertainty is relevant (i.e. affects measurements in routine testing), “real" samples must be used in the validation study. If this is not practicable (e.g. because of stability issues), and homogenous test material is used instead, then the sample preparation and/or subsampling components of repeatability must be estimated in a separate experiment.

Note: The subsampling component must not be confused with the variation of bias across matrices (matrix mismatch) component, which may vary considerably from lab to lab, thus inflating the between-laboratory component (rather than the repeatability).

In the absence of fundamental variability, a simple procedure for estimating the sample preparation and/or subsampling components is as follows: conduct an in-house experiment with 12 “real” samples (routine samples). For each sample, 2 test results are obtained under repeatability conditions. Any uncertainty from heterogeneity and/or sampling steps will manifest itsef as the “within-sample” estimate, following the evaluation scheme described in 9.1, 9.2 and 9.3 above.

Procedures for characterizing fundamental variability

Fundamental variability is a subcomponent of the repeatability error term from the basic model in Section 3 and denotes the irreducible variation between samples which remains even under the highest achievable degree of homogeneity. Fundamental variability reflects heterogeneity at the level of the sample’s constituent particles; it has an influence on the uncertainty of measurement results when the target analyte is located on sparsely distributed carrier particles[10]. Fundamental variability appears twice: first, during sampling, and second, during subsampling in the laboratory, i.e. extraction of a test portion after homogenization of the laboratory sample. In practice, nonnegligible fundamental variability can be reduced by modifying the testing procedure in two respects: first, by finer grinding or comminuting or mixing of the test material, and second, by increasing the test portion size.

It should be noted that, while a correct partitioning of observed variability between sampling, subsampling and other uncertainty components is achievable in theory, doing so is difficult in practice *when the fundamental variability is significant*. Take the case that the number of carrier particles in the laboratory sample collected from the container or batch of products varies randomly between 0 and 10. The fundamental variability between subsamples (test portions) will thus depend on which laboratory sample they were collected from. In such a situation, a correct characterization of fundamental variability would be quite involved. It would be much more efficient to ensure variation regarding carrier particle numbers between laboratory samples were negligible – in other words, to ensure that every single laboratory sample were representative of the container or batch of products, thus eliminating the sampling fundamental variability from the equation. Often, this may be achieved by increasing laboratory sample size; but a more general point is that a correct evaluation of fundamental variability requires an appropriate inclusion of the sampling step, i.e. a consideration of the different steps from sampling to analysis as one single process[11].

The question thus arises: how can we decide whether fundamental variability is significant? Fundamental variability cannot be characterized by means of classical homogeneity studies such as the standard designs described in ISO 13528 [22] and Guide 35 [9]. Indeed, in these designs, it is not possible to distinguish fundamental variability from sample heterogeneity *per se*, so that the former may be mistaken for the latter.

The following procedure allows a characterization of fundamental variability.

Step 1

Check whether one of the following criteria are met:

Criterion 1: The in-house repeatability standard deviation is larger than 3 times the expected value.

Criterion 2: The in-house repeatability standard deviation is larger than the Horwitz SD value.

Criterion 3: Conspicuous “upper” outliers are present in QC data. For instance, in the QC data provided in Table 2 (Section 9.1), the Day 7 value of 14.54 could be considered such an “upper” outlier. The presence of such outliers constitutes a further indication that the unexpectedly large observed variability may be due to fundamental variability.

If at least one of these criteria is met, proceed to Step 2.

Step 2

Conduct the following experiment:

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10 Fundamental variability is related to Pierre Gy’s fundamental error, see [23].

11 Consider the following example: a 5 t container contains one single carrier particle, translating to 1 µg/kg analyte concentration. A 5 kg laboratory sample is collected from the container. Thus, with 99.9 % probability, the laboratory sample will contain no carrier particle, and there will be no fundamental variability. However, with 0.1 % probability, the laboratory sample will contain the single carrier particle. In such a case, if a 500 g test portion is taken from the laboratory sample, then the analyte concentration in the test portion will be either 0 mg/kg (nine times out of ten) or 10 mg/kg (one time out of ten). This corresponds to a (Poisson) standard deviation of 1 mg/kg – which clearly constitutes a disproportionate estimate in relation to the situation in the container. This example shows how restricting the calculation of fundamental variability to the subsampling step can lead to gross misestimation.

1. Obtain 20 test results under repeatability conditions. Calculate the corresponding variance s_1^2.
2. Increase test portion size by a factor k (e.g. triple test portion size, $k = 3$). If it is not possible or practical to increase test portion size, grinding and homogenizing a volume corresponding to a k-fold increase in test portion size prior to taking a test portion with the original size is another option.
3. Obtain 20 test results under repeatability conditions on the basis of the finely ground test material / increased test portion size. Calculate the corresponding variance s_2^2.
4. If the ratio $\frac{s_1^2}{s_2^2}$ is greater than 2.17, then calculate the SD characterizing fundamental variability as follows:

$$s_F = \sqrt{\frac{k}{(k-1)} \cdot (s_1^2 - s_2^2)}$$

5. If the ratio $\frac{s_1^2}{s_2^2}$ is less than 2.17, this is an indication that fundamental variability is not significant and does not need to be included in the estimate of measurement uncertainty.

Example

Table 9: Test results from an experiment for the calculation of fundamental variability

	Experiment 1: Original test portion size	Experiment 2: Test portion size is tripled
Sample 1	14.0	15.1
Sample 2	11.9	13.8
Sample 3	10.5	11.8
Sample 4	14.9	14.0
Sample 5	13.1	11.4
Sample 6	9.5	15.7
Sample 7	15.6	12.4
Sample 8	18.3	11.5
Sample 9	12.5	12.1
Sample 10	16.4	13.7
Sample 11	18.0	15.8
Sample 12	14.0	12.5
Sample 13	13.0	12.8
Sample 14	20.8	15.1
Sample 15	10.2	11.8
Sample 16	21.5	10.6
Sample 17	13.9	11.1
Sample 18	17.8	12.9
Sample 19	7.7	11.4
Sample 20	12.2	16.3

Note that, in Experiment 1, several conspicuously large values are obtained – an indication that fundamental variability is non-negligible.

The following variances and corresponding ratio are obtained:

Table 10: Variances and their ratio

s_1^2	s_2^2	s_1^2/s_2^2
13.54	3.05	4.44

As can be seen, the ratio s_1^2/s_2^2 is greater than the value 2.17. Accordingly, the fundamental variability is calculated as

$$s_F = \sqrt{\frac{3}{2} \cdot (s_1^2 - s_2^2)} = 3.97.$$

9.5 PROCEDURES FOR DETERMINING WHETHER THE ESTIMATE OF MEASUREMENT UNCERTAINTY DERIVED FROM PRECISION DATA FROM A COLLABORATIVE STUDY IS COMPLETE

In general, the workload associated with a *reliable* quantitative assessment whether a measurement uncertainty estimate is complete will prove prohibitive. For this reason, a qualitative assessment is proposed, consisting in addressing the two following questions.

Are uncertainty contributions from subsampling and sample preparation included in the repeatability estimate and/or reproducibility estimate?

Whether or not subsampling is included depends on the sample material provided to the laboratories. If the material is grinded/homogenized, subsampling uncertainty is not included.

Whether or not sample preparation is completely included depends on which sample preparation steps have already been performed by the organizer of the collaborative study.

If these sources of uncertainty were not duly reflected in the collaboratorive study, additional experiments as described in Section 9.4 are required.

Are matrix mismatch effects reflected in the reproducibility estimate?

In general, this is not the case. The influence of matrix mismatch can only be determined via comparison with reference values or via spiking experiments, see the procedure proposed in Section 9.2. Another example can be found in Annex F of ISO TS 23471 [20], where equipment and operator effects along with matrix mismatch effects arising from different lots and storage conditions are analyzed by means of a factorial design.

10 Influence of measurement uncertainty on sampling plans: examples

In the *General Guidelines on Sampling* [13], it is stated that “Codex Methods of Sampling are designed to ensure that fair and valid sampling procedures are used when food is being tested for compliance with a particular Codex commodity standard”. Sample size and acceptance number / acceptability constant for inspection by attributes / variables are determined on the basis of procedures and sampling plans described in ISO standards and/or Codex guidelines. While measurement uncertainty may be considered irrelevant for inspection by attributes, its impact on inspection by variables must be accounted for.

In the introduction to ISO 3951-1:2013, it is stated that “[i]t is assumed in the body of this part of ISO 3951 that measurement error is negligible […]”. Nonetheless, procedures for increasing the sample size are provided in Annex B of ISO 3951-1 [14] and Annex P of ISO 3951-2 [15] for the case that measurement uncertainty is non-negligible. It is important to note that these procedures are only applicable if “the measurement method is unbiased, i.e. the expected value of the measurement error is zero” (see Annex P.1 in ISO 3951-2:2013 [15]). In such a case, total variability is expressed as

$$\sigma_{total} = \sqrt{\sigma^2 + \sigma_m^2}$$

where σ denotes the process standard deviation and σ_m denotes the measurement standard deviation.

If σ_m is non-negligible (i.e. greater than one tenth of the sampling standard deviation s or process standard deviation σ), the sample size n must be increased to either $n^* = n \cdot (1 + \gamma^2)$ where $\gamma = \sigma_m/\sigma$ (the process standard deviation σ is known) or $n^* = n \cdot (1 + \tilde{\gamma}^2)$ where $\tilde{\gamma}$ is an estimated upper bound of $\gamma = \sigma_m/\sigma$ (the process standard deviation σ is unknown). The acceptability constant k remains unchanged. For further details, see Annex P in ISO 3951-2:2013 [15].

Example

A lot of 500 items of pre-packaged mineral water is assessed for sodium content. If the measurement uncertainty is not taken into consideration, for an agreed AQL of 2.5 % (maximum concentration 200 mg/L), general inspection level II (default level) a sample of 30 items should be collected for assessment, (ISO 3951-2 [15], Annex A, Table A1 and Annex B, Table B1). The production is well under control and the control charts give a process standard deviation σ of 2 mg/L. The measurement uncertainty standard deviation σ_m is 1 mg/L and is thus non-negligible. With $\gamma = \sigma_m/\sigma = 0.5$ and $1 + \gamma^2 = 1.25$ the sample size must be increased to 38.

If there is a bias, the above procedure must be modified. One possibility would be to proceed as follows[12]. The standard deviation of $\bar{x}$, the mean across the n measurement results, is expressed as

$$\sigma_{\bar{x}} = \sqrt{\frac{\sigma^2 + \sigma_0^2}{n} + \sigma_b^2}$$

where σ denotes the process standard deviation, σ_0 denotes the repeatability component of measurement uncertainty (calculated on the basis of the n items sampled from the lot), and σ_b represents available information (e.g. the between-lab standard deviation from a method validation study) used to estimate the bias term.

The modified procedure is as follows:

1. Increase the sample size under the assumption that there is no measurement error
2. Calculate $d = \frac{1}{n} - \frac{\sigma_b^2}{\sigma^2}$
3. If $d \leq 0$, inflated variability due to a bias cannot be compensated for via an increase in sample size.
4. If $d \leq \frac{1}{2n}$, bias compensation via an increase in sample size may not be appropriate due to the large number of samples required. It is then suggested to reduce bias or to use another measurement method.
5. If $d > \frac{1}{2n}$, calculate the new sample size as $n^* = \frac{1+\frac{\sigma_0^2}{\sigma^2}}{d} = \frac{\sigma^2+\sigma_0^2}{\frac{\sigma^2}{n}-\sigma_b^2}$

Example (continued from previous example)

It is now assumed that there is a method bias and that a σ_b estimate of 0.2 mg/L is available. Accordingly, on the basis of the previously calculated value of $n = 38$, d is calculated as $d = 0.016$. Since $d > \frac{1}{2n} = 0.013$, the new sample size is calculated as $n^* = 77$ (with $\sigma_0 = \sigma_m = 1$ mg/L).

Procedures for bulk sampling

Procedures for bulk sampling are provided in ISO 10725:2000 [17]. As in the case of sampling from packages, these procedures are only valid under the assumption that there is no method bias. Modified procedures for the case that there is a method bias are currently being developed. For now, the discussion is limited to the case that there is no bias.

A *dominant* measurement uncertainty has an effect on the number of test samples per composite sample n_T as well as the number of measurements per test sample n_M. The measurement uncertainty is dominant when both the standard deviation of the sampling increment σ_I and the standard deviation between test samples σ_P are far less (one tenth or less) than the measurement standard deviation σ_M (i.e. the measurement uncertainty), which must be known and stable, see Annex B in ISO 10725 [17]. The number of sample increments per composite sample n_I remains unchanged, no matter whether the measurement uncertainty is dominant or not. The mass of the increments should be sufficiently large to offset the fundamental variability.

Example

A lot of wheat bulk material is to be assessed for cadmium content (maximum concentration e.g. 0.1 mg/kg). In this example, it is assumed that cadmium concentrations in the lot are homogeneous, resulting in very low standard deviations σ_I and σ_P, estimated as 0.0015 mg/kg and 0.002 mg/kg, respectively. Since the concentrations are very low, a relatively high measurement uncertainty $\sigma_M = 0.025$ mg/kg is obtained. The discrimination interval D (difference between agreed risk-based acceptance and rejection levels) is 0.02 mg/kg.

[12] This modified procedure is taken from the current stage of development of Annex B of ISO/DIS ISO 3951-6 [16].

The measurement standard deviation $\sigma_M = 0.025$ mg/kg is thus dominant (d_I is calculated as 0.075). The number of increments per composite sample is $n_I = 6$, the number of test samples per composite sample is $n_T = 2$ and the number of measurements per test sample is $n_M = 2$ (yielding a product $n_T \cdot n_M = 4$, which can be interpreted as a measure of the analytical workload). The combined overall standard deviation σ_0 is calculated as $\sqrt{\frac{n_T \cdot n_M}{n_I}\sigma_I{}^2 + n_M\sigma_P{}^2 + \sigma_M{}^2} \approx 0.03$ mg/kg and divided by the discrimination interval D in order to obtain the relative standard deviation $d_0 = \sigma_0/D \approx 1.26$. By means of Table B1 in Annex B of ISO 10725 [17], this relative standard deviation d_0 is used to determine the adjusted number of test samples per composite sample $n_T = 2$ (i.e. n_T remains the same) as well as the adjusted number of measurements per test sample $n_M = 3$, yielding a product $n_T \cdot n_M = 6$.

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APPENDIX IV

Revised *General Guidelines on Sampling* (CXG 50-2004)

(for adoption at Step 8)

1 Reference guidelines

1.1 Introduction

The guidelines are primarily intended for use by Codex commodity committees responsible for developing acceptance sampling plans for provisions in Codex standards, and by governments responsible for import or export inspection of foods to describe the design and evaluation of sampling plans for the international trade of food commodities.

Foods are frequently sampled, throughout the food supply chain from producers to consumers, for the purpose of checking their quality. Clear definition of sampling plans is an integral part of specifications for the sampling and testing of foods. Sampling plans are included in Codex standards and may be used by governments in standards for foods.

Codex sampling plans, in conjunction with methods of analysis, are intended as a means of verifying that foods comply with provisions relating to composition, chemical or microbiological contaminants or pesticide residues contained in Codex standards.

Sampling therefore plays an important role in achieving the Codex objectives of protecting consumers' health and ensuring fair practices in the food trade. Codex sampling plans also have an important role in harmonizing technical approaches to sampling and by results of analysis interpretation in relation to lots or consignments of foods, in the light of the relevant provision(s) of the applicable Codex standard.

It is important that sampling be undertaken in a way that contributes to these objectives.

Specification of these quality objectives, the quality level acceptable to the customer and the rate of acceptance of compliant products, enables the development of sampling plans.

A Codex standard may set out a specific sampling plan for a particular context, or it may specify the outcome to be achieved by a sampling plan.

Although these guidelines provide a generic approach to the design of sampling plans, Codex sampling plans are intended primarily for inspection of foods upon receipt, for example by importing country regulatory agencies, and might not be suitable for use by producers. However, a clear definition of quality objectives in Codex standards will allow producers to devise appropriate control and inspection procedures to achieve them.

1.2 Scope

In these guidelines, the focus is on acceptance sampling plans for the inspection of isolated homogeneous lots, in which the risks to consumers and producers are controlled. Additionally, there are some guidelines for sampling inhomogeneous lots.

The term 'isolated' means that the inspection of each lot is done in isolation, without considering the outcome of the inspection of adjacent lots or, for example, other lots from the same producer. This does not mean that information from previous inspections cannot be used; in particular, there are cases where the lot standard deviation may be known from the inspection of previous lots.

The following situations are covered:

- acceptance sampling plans for the control of the percentage nonconforming for homogeneous lots by attributes or by variables, for goods in bulk or individual items;
- inspection by variables sampling plans for normally distributed characteristics;
- adjustment for measurement uncertainty in cases where it is non-negligible as compared to the lot standard deviation with a focus on cases where the measurement uncertainty is normally distributed;
- sampling plans for the control of the average content; and
- in addition, some information is provided on issues involved with the design of plans for bulk materials.

In section 2, general concepts which are relevant for the sampling of foods are defined, sections 3, 4 and 5 cover acceptance sampling plans for different situations of statistical food control. Section 6 covers other matters such as physical sampling and inhomogeneous lots.

Appendix I contains a step-by-step guide for the selection of sampling plans. Appendix II contains tables of ISO[i] attributes and variables plans indexed by producer's risk.

These guidelines are not intended to be comprehensive; these guidelines do not provide information on all types of sampling plan options that may be available. Sampling plans from other sources, such as plans developed by other Codex committees, are still acceptable subject to their endorsement by the Codex Committee of Methods of Analysis and Sampling (CCMAS).

1.3 Definitions

For the terms commonly used in these guidelines, the following definitions are provided, in addition to those in the *Guidelines on Analytical Terminology* (CXG 72-2009).[1]

Note: In some of the definitions, reference is made to the process standard deviation or the process quality level. In these guidelines, the focus lies on lots rather than processes. For this reason, the relevant quantities in these guidelines are the lot standard deviation and the lot quality level.

Acceptance criterion

Acceptance criterion is used to cover terms such as acceptance and rejection numbers for attributes plans and acceptability constants for variables plans.

Note: In these guidelines, the term 'acceptance criterion' is used to describe the rule which is applied to the test results obtained during the lot inspection in the decision whether to accept the lot.

Acceptance sampling

Sampling after which decisions are made to accept a lot, or other grouping of products, materials, or services, based on sample results.

Acceptance sampling plan

Plan which states the sample size(s) to be used and the associated criteria for lot acceptance.

Acceptance sampling by attributes

Acceptance sampling inspection whereby the presence or absence of one or more specified characteristics of each item in a sample is observed to establish statistically the acceptability of a lot or process.

Acceptance sampling by variables

Acceptance sampling inspection in which the acceptability of a process is determined statistically from measurements on specified quality characteristics of each item in a sample from a lot.

Conformity assessment

Activity to determine whether specified requirements relating to a product, process, system or person or body are fulfilled.

Consignment

A quantity of some commodity delivered at one time. It may consist of either a portion of a lot, or a set of several lots.

However, in the case of statistical inspection, the consignment shall be considered as a new lot for the interpretation of the results.

- If a consignment is a portion of a lot, the consignment shall be considered as a lot for the inspection.
- If the consignment is a set of several lots, before any inspection, care shall be given to the homogeneity of the consignment. If not homogeneous, a stratified sample may be used.

Consumer and producer

The terms 'consumer' and 'producer' are conventional and may apply to a range of different operators in the food supply chain, such as a grower, manufacturer, the manufacturer's own quality control system, supplier, exporting country, processor, on-seller, or importing country. In general, 'producer' refers to a supplier or seller of foodstuffs and 'consumer' to an importing country regulator, a purchaser, or an actual consumer of those foods.

[i] The International Organization for Standardization (ISO).

Consumer's risk (CR)

Probability of acceptance when the quality level of the process has a value stated by the acceptance sampling plan as unsatisfactory.

Consumer's risk quality (CRQ)

Quality level of a lot or process which, in the acceptance sampling plan, corresponds to a specified CR.

Note: The CRQ corresponds to the LQL in the ISO 2859[2] and ISO 3951[3] standards.

Indifference quality level

Quality level which, in the acceptance sampling plan, corresponds to a probability of acceptance of 0.5, when a continuing series of lots is considered.

Laboratory sample

A sample as prepared (from the lot) for sending to the laboratory and intended for inspection or testing.

Lot

A quantity of product produced under conditions presumed uniform.

Operating characteristic curve

Curve showing the relationship between probability of acceptance of product and the incoming quality level for given acceptance sampling plan.

Plan

Refer acceptance sampling plan.

Producer's risk (PR)

Probability of non-acceptance when the quality level of the process has a value stated by the plan as acceptable.

Producer's risk quality (PRQ)

Quality level of a lot or process which, in the acceptance sampling plan, corresponds to a specified PR.

Note: The PRQ corresponds to the AQL in the ISO 2859[2] and ISO 3951[3] standards.

Quality level

Quality expressed as a rate of nonconforming units or rate of number of nonconformities.

Note: In these guidelines, the quality level of a given lot is often expressed in terms of the percentage of nonconforming items.

Sample

One or more items taken from a population and intended to provide information on the population, and possibly serve as a basis for a decision on the population or on the process which had produced it.

Sampling plan

Refer acceptance sampling plan

2 Acceptance sampling – general principles

2.1 Reasons for sampling

While various measures such as hazard analysis and critical control point systems (HACCP), good manufacturing practice (GMP), process control and sampling are available to producers to provide assurance about the quality of products they supply, consumers usually rely on acceptance sampling if they wish to verify the quality of incoming products.

Acceptance sampling procedures are used when goods are transferred between two parties. The purpose of these procedures is to provide unambiguous rules for releasing a product after inspection of only a limited sample. Both parties should be fully aware of the limitations and risks associated with using such procedures and therefore most acceptance sampling procedures should include provisions for dealing with disputes and non-conforming items found in lots that have been accepted by the sampling plan.

An acceptance sampling plan specifies the number of items to be taken and how they are to be taken, the acceptance criterion used to decide whether a lot should be accepted and how to take non-negligible measurement uncertainty into account.

In general acceptance sampling is used to:

- reduce costs;
- allow product assessment when tests are destructive; and
- enable faster decision-making.

2.2 Approaches to acceptance sampling

There are three possible approaches to acceptance sampling:

(a) 100 percent inspection, involving inspection of the entire (i.e.100 percent) lot;

(b) sampling based on statistical principles; and

(c) ad hoc inspection, i.e. sampling plans without a statistical basis.

The risks and costs associated with each of these three options will be briefly discussed. Approach (a) is usually not feasible due to the prohibitive cost of testing and in addition, there might not be any product left to sell if the inspection method necessitates destructive testing.

Approach (b) has the disadvantage of higher risks as compared to approach (a), since a part of the lot is not inspected. However, by applying an approach based on statistical principles, the risks can be calculated, and a sampling plan can be chosen that ensures these risks are controlled to desired levels. It also has the advantage of practicability and lower costs.

In lot inspections, there are two types of risks:

- acceptance of a lot of unsatisfactory quality (CR); and
- rejection of a lot of acceptable quality (producer's risk).

Sampling plans should be designed to control these risks to suitable levels, whereby suitable risk levels are determined based on fitness for purpose considerations.

Approach (c) is not recommended. It may be used for practical reasons, such as limited resources, or for simplicity. However, such plans might not provide the expected level of assurance of food quality and may inadvertently impose high costs, for instance through unwarranted acceptance of food that could lead to illness or unjustified rejection that, in turn, could lead to the imposition of fines, penalties or trade sanctions. The risks associated with such plans should be evaluated where possible. Decisions on acceptance or rejection should not be made solely based on these plans except by mutual agreement of the consumer and producer with an understanding of the risks involved.

In summary, approach (b) allows for practicability while ensuring that risks are controlled to levels considered appropriate based on fitness for purpose considerations.

2.2.1 **Acceptance sampling versus conformity assessment**

Acceptance sampling and conformity assessment do not have the same purpose. Conformity assessment is the use of a single measurement result to decide whether a single item conforms to a limit. Acceptance sampling is the process in which a sample[ii] is taken from a lot and involves the determination of acceptance criteria and sample size to decide whether a lot is accepted or rejected.

The broadest definition of conformity assessment may be considered to include acceptance sampling. However, in a narrower sense, conformity assessment can be understood to refer specifically to the situation where a one single measurement result is used to decide if one single item of interest conforms to a specified requirement. If conformity assessment is understood in this narrower sense, then it is important to distinguish conformity assessment and acceptance sampling. In this section, conformity assessment will be understood in the narrower sense.

Although acceptance sampling and conformity assessment involve similar procedures, and although consumer and producer risks are defined for both, they are performed in different contexts and follow different objectives.

Conformity assessment

In conformity assessment, conformity is assessed via the application of a decision rule which accounts for measurement uncertainty. Depending on the measurand, the measurement uncertainty may or may not include uncertainty from sampling. Depending on the decision rule, there may be cases where the assessment is inconclusive.

Acceptance sampling

In acceptance sampling, at least one measurement result (typically more than one) is used to decide whether to accept or reject a lot under inspection. The acceptance sampling plan consists in both requirements regarding the sampling procedure (e.g. the number of items to be taken from the lot) and an acceptance criterion. The acceptance sampling plan is determined in such a way as to ensure that producer and/or consumer risks are sufficiently low at a given quality level. The variation of the property of interest in the lot is always taken into consideration in acceptance sampling; however, analytical uncertainty is only taken into consideration if non-negligible. The context for lot inspection is typically a commercial agreement between two trading partners. In acceptance sampling, a lot is always either accepted or rejected; there are no cases of inconclusive lot inspections.

In the case that the quality level is expressed in terms of the percentage of nonconforming items, the distinction between acceptance sampling and conformity assessment is quite clear; the measurand is defined for the individual items, and thus the question of conformity to a specified requirement can only be framed in relation to the individual items. However, lot acceptance or rejection is not decided on the basis of the compliance or non-compliance of an individual item. Instead, the acceptance criterion is expressed in terms of the percentage of nonconforming items, in terms of the distribution of the property of interest among the items in the lot. The differences between acceptance sampling and conformity assessment are summarized in the following table.

[ii] Refer to the definition in section 1.3.

Table 1: Differences between acceptance sampling and conformity assessment

	Conformity assessment	Acceptance sampling
Number of measurement results	Typically: one	Typically: several (For instance: if the lot consists of discrete items, several items are taken, and there is one measurement result per item)
Is analytical measurement uncertainty taken into account in the decision rule/acceptance criterion?	Always (if possible)	Only if the analytical measurement uncertainty is non-negligible (compared to the lot standard deviation)
Are any components of sampling uncertainty considered?	Depending on the measurand, it may or may not be necessary to include sampling uncertainty	The variation of the characteristic of interest within the lot is considered via the lot standard deviation
Context/background	In many cases: conformity assessment is carried out against a legal limit	The context is often an agreement between trading partners
Inconclusive assessment	Depending on the decision rule, the assessment may be inconclusive	There are no inconclusive inspections: lots are either accepted or rejected.

Further clarifications regarding the term measurand and the distinction between sampling and analytical uncertainty are provided in section 5.2.1.

Note 1: Figure 1 in the Guidelines on Measurement Uncertainty (CXG 54-2004)[4] illustrates a procedure which can be applied in conformity assessment (this procedure may yield inconclusive results). This procedure should not be applied in acceptance sampling.

Note 2: If the sample taken in a lot inspection consists of one single item, then producer/consumer risks may be poorly controlled. Nonetheless, there are special sampling plans for lot inspection based on a single item. These must not be confused with the procedure for conformity assessment illustrated in Figure 1 of in the Guidelines on Measurement Uncertainty (CXG 54-2004).[4]

2.3 Acceptance sampling plan performance

Variation is present everywhere; raw materials vary in their composition, manufacturing processes vary and, consequently, the products manufactured by those processes will also vary. Therefore, when we take several samples from a lot, we do not expect those samples to be of the same composition. Furthermore, the presence of measurement uncertainty means that when those samples are tested, we will not get the same result, even if the same sample is retested. Similarly, we would not expect results from different sets of samples taken from the same lot or those taken from different lots (from the same process) to be the same; there will always be some variation.

Due to this variation, the incorrect acceptance or rejection of lots cannot be avoided. However, using a statistical description of the variation within a lot and of the uncertainty of the measurement process allows us to calculate the probability of correctly or incorrectly accepting a lot at any given quality level and for any given sampling plan.

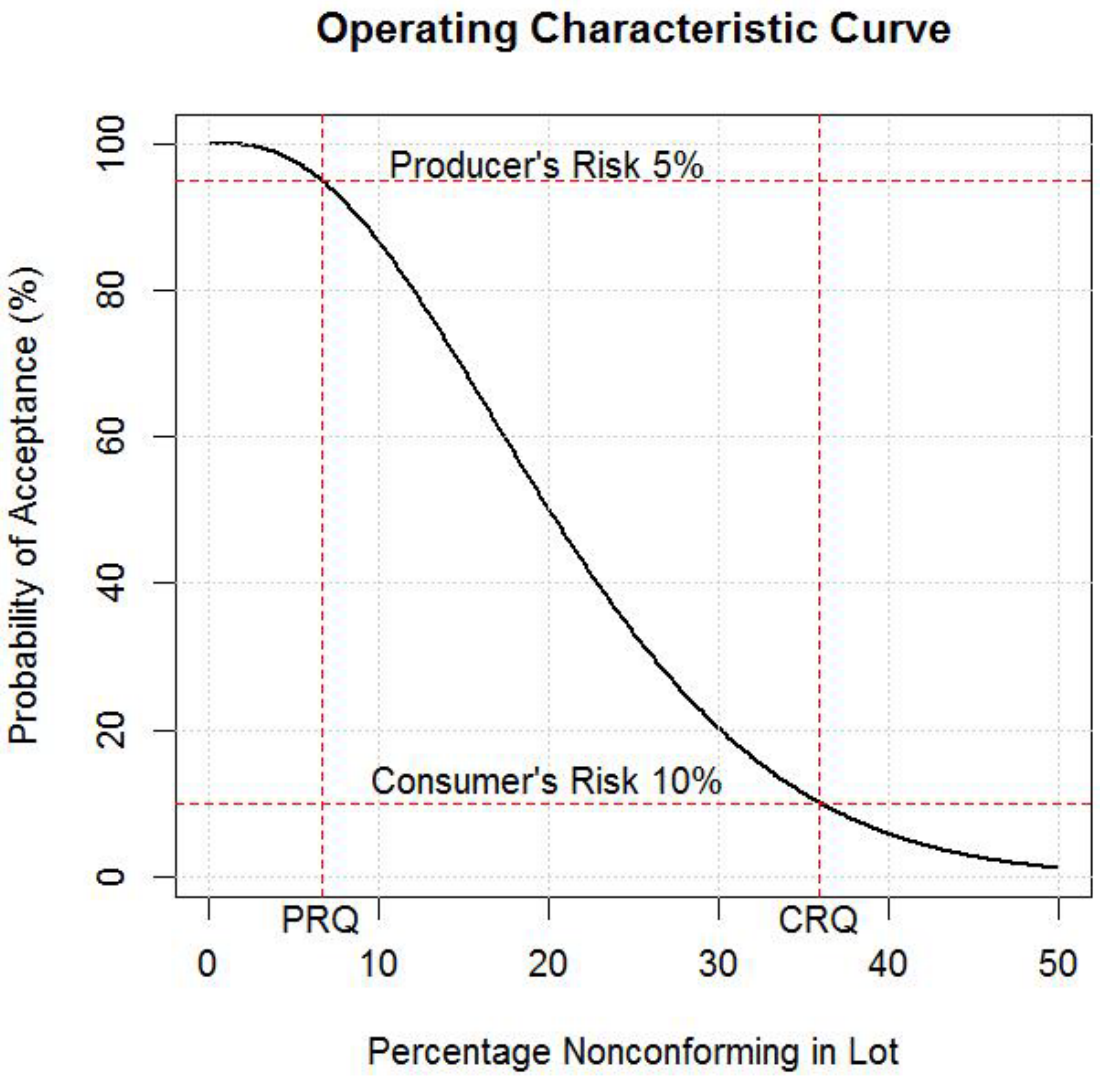
In acceptance sampling, the probability of acceptance depends on:

- the quality level (percent nonconforming) of the lot under inspection;
- the acceptance criterion (i.e. for the particular sampling plan);
- the variation of the characteristic within the lot; and
- the bias and variation inherent in the measurement process (in the case of non-negligible analytical uncertainty).

In practice, the quality level (percent nonconforming) of a lot is not known beforehand; however, for a particular acceptance sampling plan, it is possible to calculate the probability of acceptance at any quality level. The relationship between the probability of acceptance and the quality level for a particular sampling plan is described by the operating characteristic curve.

2.3.1 Operating characteristic curve

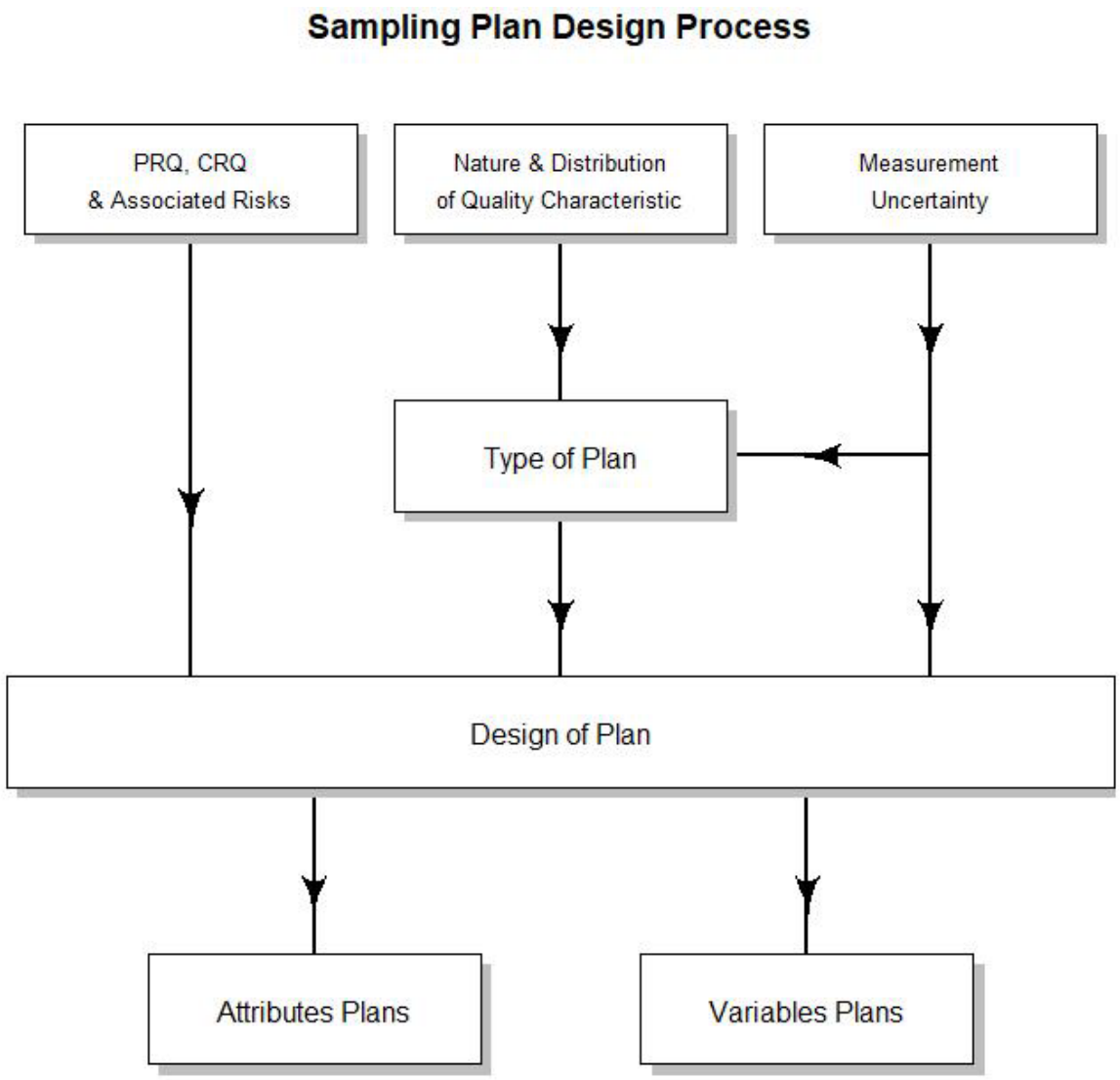
The following diagram is an example of an operating characteristic curve (OC curve) that shows the probability of accepting (or rejecting) a lot in terms of its quality level in the lot (expressed as percent nonconforming). This highlights that specification of the quality levels is fundamental to design of a sampling plan.



Note: The OC curve does not say anything about the quality of a given lot; it serves only to show the probability of accepting a lot with a particular quality level.

3 Design of sampling plans

3.1 Sampling plan design process



3.2 Inputs to sampling plans

3.2.1 Stringency

As explained, the application of acceptance sampling plans does not eliminate the risk that a lot of poor quality will be incorrectly accepted nor that a lot of good quality will be incorrectly rejected.

However, designing such plans using statistical principles allows these risks to be controlled. This is achieved by specifying a particular PRQ level, and a particular CRQ quality level, along with a corresponding PR and a CR respectively. Once these four parameters, the (PRQ, CRQ, PR and CR), are specified the probability of acceptance and therefore the producer's and consumer's risks at any quality level are uniquely determined.

The term stringency is used in these guidelines to refer to the ability of a sampling plan to control consumer's and producer's risks, of incorrectly accepting or incorrectly rejecting a lot, at any specified quality level.

Often, the PR is specified as 5 percent, meaning that the probability of rejecting a lot with PRQ is at most 5. Similarly, the consumer's risk is typically chosen as 10 percent, meaning that the probability of accepting a lot with CRQ is at most 10 percent. If any one of the four parameters is altered, the control of the producer's and consumer's risks will change.

In certain situations, such as characteristics relating to food safety where control of the CR is paramount, it might not be appropriate to take account of the PR in the design of sampling plans. This leads to two different options for the specification of risks.

Option 1: Plans that explicitly control both the CR and the producer's risk:

- both the PRQ and CRQ, along with the respective allowable probabilities of incorrect rejection (PR) and incorrect acceptance (CR) are specified.

Option 2: Plans that explicitly control only the consumer's risk:

- plans for assessments of lots consisting of discrete items.

3.2.2 Fitness for purpose

Codex methods of sampling should be *'designed to ensure that fair and valid sampling procedures are used when food is being tested for compliance with a particular Codex commodity standard'.*[iii] When commodity committees have included sampling plans in a Codex commodity standard, these should be referred to CCMAS for endorsement along with relevant information relating to the sampling plan.

Sampling plans from other sources are still acceptable subject to their endorsement by CCMAS.

The *Principles for the Use of Sampling and Testing in International Food Trade* (CXG 83-2013)[5] states:

'Sampling and testing procedures are fit for purpose in a given product assessment, if, when used in conjunction with appropriate acceptance criteria, they have acceptable probabilities of wrongly accepting or wrongly rejecting a lot or consignment'.

Fairness

With regard to fairness, consideration of both the CR and the PR is necessary to avoid situations such as the following:

- sampling plans having inappropriate stringency, e.g. plans for the assessment of composition that are more stringent than for food safety;
- high producer or consumer risks that may arise due to the use of sampling plans not based on appropriate specifications of allowable risks; and
- sampling plans not based on statistically valid principles, e.g. ad hoc plans or plans that do not (properly) allow for measurement uncertainty.

In addition, in the interests of fairness, designers of plans should also take account of the measures that the producer may have to take to ensure compliance, given that it is usually not suitable for the producer to use the same sampling plan as that used by the consumer.

In selecting a sampling plan, it should be ensured that producers are not exposed to unreasonable costs in terms of sampling and testing, loss of yields, or excessive rejection of their products to achieve compliance.

Practicality

It is important to ensure that any sampling plan chosen will be practical to apply in terms of cost of sampling and testing and ease of use.

Other strategies could be used to develop sampling plans that are more economical in terms of sampling and testing, such as:

- managing average non-compliance rates over the medium to long term, rather than possibly paying a high premium in terms of testing costs for high levels of assurance on a lot-by-lot basis;
- the use of 'indifference' plans that are designed around the 'indifference quality level' (IQL), the level of defects at which there is 50 percent acceptance, rather than based on PRQ and CRQ. This leads to plans having more manageable sample sizes; and
- offsets, sometimes called guard-bands or buffers, between the limits used in the acceptance criteria and the actual specification limits for a provision can be used to reduce CR and to mitigate possibly unreasonably high sample numbers. However, offsets should be used with caution in the interest of fairness to producers.

[iii] Section 2: Elaboration of Codex Texts: Principles for the Establishment or Selection of Codex Sampling Procedures: Purpose of Codex Methods of Sampling (Codex Procedural Manual, latest edition).

3.2.3 Specification limits

For a given characteristic, a specification limit may be expressed as a minimum or a maximum limit (or both) applied either to each individual item in a lot, or to the average level.

Specification limits should apply to the 'true' values of the characteristics rather than to the measurements themselves. It follows that the assessments of lot compliance should also be in terms of the 'true' values of the characteristic within the lot (see section 5.2.1).

Offsets

It is important to consider whether a given specification limit has an in-built offset (guard-band), and whether the offset reflects the measurement uncertainty associated with a particular sampling plan, that might include both analytical and sampling uncertainties.

Many provisions for chemical and microbiological contaminants have in-built offsets between the specification limits and the levels of contamination at which foods might become unsafe to consume. In such cases one may not need to design plans to provide high levels of protection against exceeding the limits as the CR is already well controlled by these offsets.

The use of offsets enables a reduction in sample size; for example, while large sample sizes are needed to show that a lot contains no more than say 1 percent nonconforming product, much smaller sample sizes are required to show that no more than 10 percent of the product in a lot exceeds a tightened limit.

3.2.4 Lot homogeneity

Acceptance sampling plans are usually based on the assumption that lots are homogeneous; indeed, the international definition of a lot is 'a quantity of product produced under conditions presumed uniform'.

In these guidelines, the term 'homogeneous' does not mean that the characteristic of interest does not vary within the lot. Rather, the term 'homogeneous' means that it is possible to characterize the variation of the characteristic of interest within the lot by means of a single standard deviation. Homogeneity applies only to variables plans.

In considering homogeneity, one needs to draw a distinction between:

- the type (shape) of the distribution, (e.g. *normal* distribution); and
- the *spatial distribution* of the characteristic within the lot.

If the lot consists of discrete items and if random sampling is used (as recommended for all plans in these guidelines), then the spatial distribution does not matter and the lot cannot always be considered homogeneous.

For this reason, if no prior information regarding the spatial distribution is available, then random sampling should be performed.

On the other hand, if prior knowledge indicates that the spatial distribution of the characteristic within the lot is random, then random sampling is not required. This case corresponds perhaps to the intuitive understanding of what homogeneity means in the context of acceptance sampling.

If random sampling cannot be performed, then the lot can only be considered homogeneous to the extent that the spatial distribution is random. In this sense, if random sampling cannot be performed, the homogeneity of the lot depends on the spatial distribution.

For some lots consisting of bulk material, inhomogeneity means that several segments must be sampled from.

Sections 4.4 and 6.3 provide further guidance regarding the inspection of inhomogeneous lots consisting of bulk materials or discrete items, respectively.

3.2.5 Distribution of the characteristic

The options for sampling plans depend on whether the test results are measurements (variables data) or have nominal outcomes (attributes data). In some cases, variables data can be classified as binary outcomes, but this should only be done after careful consideration of the sampling options available as the sample size for attributes inspection can be much larger than for variables data.

In the case of variables data, the assumed statistical distribution of the measurements in the lot should also be specified, i.e. whether the characteristic is normally distributed, a compositional proportion, or follows some other distribution. If is not possible to make an assumption regarding the distribution of the data, results can be classified as attributes (as long as measurement uncertainty is negligible [refer section 3.2.8]), or plans based on the fractional nonconformance (FNC) method can be used (as long as measurement uncertainty is non-negligible [refer section 5.2.6]).

However, the characteristic does not have to follow the assumed distribution exactly (and, in any case, it is difficult to verify conformance to a distribution based on a small sample size). In practice, it is sufficient that the assumed distribution provides a satisfactory model for the behaviour of the characteristic in the lot. However, if the actual distribution in the lot differs markedly from the assumed distribution, then the producer's and consumer's risks may exceed the allowed levels specified in the design of the plan.

A typical 'default' assumption in variables plans is that the characteristic follows a normal assumption.

It is important to note that in the case of attributes plans, the binomial distribution is always available as 'default' assumption, and that departures from this assumption regarding the type (shape) of the distribution will have very little impact on the producer's and consumer's risks.

Sections 4.4 and 6.3 provide further guidance regarding the inspection of inhomogeneous lots consisting of bulk materials or discrete items, respectively.

Prior knowledge of the distribution of a characteristic

In acceptance sampling, acceptance/rejection of a lot is decided on the basis of a sample (the set of individual items or increments taken from the lot). The relationship between the probability of acceptance (upon application of a given sampling plan) and the quality level of the lot is determined on the basis of prior knowledge regarding the distribution of the characteristic within the lot.

This means that prior knowledge is required *even in connection with the inspection of isolated lots.* In other words, the inspection of isolated lots does not mean that no prior information is available. On the contrary, prior information is always required. Sometimes the prior information takes the form of (tacit) assumptions based on experience and expert judgement. For example, a typical 'default' assumption in variables plans is that a characteristic follows a normal distribution.

If the actual distribution in the lot differs markedly from the assumed distribution, then the producer's and consumer's risks may exceed the allowed levels specified in the design of the plan. There are two ways in which the actual distribution can differ from the distribution which was assumed on the basis of prior knowledge:

- the type (shape) of the distribution. For example, the assumption is that the distribution is normal whereas, in fact, the distribution is lognormal; and
- the parameters of the distribution. For example, it is assumed that the lot standard deviation is the same as the (underlying) process standard deviation, whereas in fact it is twice as large.

It is important to note that in the case of attributes plans, the binomial distribution is always available as 'default' assumption, and that departures from this assumption regarding the type (shape) of the distribution will have very little impact on the producer's and consumer's risks.

3.2.6 Lot standard deviation

In the context of these guidelines, the population under consideration is the lot itself rather than the underlying process. For this reason, the role which the *process* standard deviation σ plays in the ISO 3951**Error! Bookmark not defined.** standards is now played by the lot standard deviation. The lot standard deviation can be represented by either its true value σ (sigma) or by an estimate (often denoted s) of σ.

The lot standard deviation is relevant only for variables plans, particularly for characteristics that are normally distributed or follow distributions, such as the lognormal distribution,[iv] that are related to the normal distribution.

For a given characteristic, the lot standard deviation is a measure of the random variation of the characteristic within the lot under inspection. Its estimate, however, may be affected by components of analytical or sampling uncertainty.

It is expected that for isolated lots the lot standard deviation will usually be calculated from the test results obtained during the inspection. Notwithstanding, there are cases where the lot standard deviation may be known, especially when the lot has been produced by a process with a known process standard deviation. This can be adopted as lot standard deviation. In such cases, the sample size of the sampling plan can be considerably reduced.

If the process standard deviation is known, it is important to consider whether it was obtained on the basis of a sufficiently large number of data to ensure it provides a reliable characterization of the variation within the process.

[iv] For lognormally-distributed characteristics, the logarithms of the 'measurements' are normally distributed.

Note: In acceptance sampling, the lot standard deviation is always based on a simple random sample. However, in principle, other sampling procedures may be applicable, such as those described in Annex C.2 of the EURACHEM[v]/CITAC guide to measurement uncertainty arising from sampling.[6] This guide describes several procedures for the calculation of sampling uncertainty. It does not describe procedures for acceptance sampling.

3.2.7 Measurement uncertainty

In connection with lot inspections, it is important to determine whether the analytical components of measurement uncertainty – including the uncertainty which arises from subsampling from the laboratory sample (see section 5.2.6) – can be considered negligible. This is typically done by considering the ratio of the analytical uncertainty and the lot standard deviation. If the analytical component of measurement uncertainty cannot be considered negligible, it should be taken into consideration in the acceptance criterion.

Adjustment for the analytical component of measurement uncertainty in acceptance sampling is discussed in more detail in section 5.

The lot standard deviation already represents the variation of the characteristic of interest within the lot and any further uncertainty arising from the sampling procedure. For this reason, in determining whether an adjustment is necessary, only the analytical component of measurement uncertainty needs to be considered.

The term *measurement error* should not be used, as the term has been superseded by the focus on uncertainty across JCGM,[vi] ISO and EURACHEM[vii] standards and guides, as reflected in the *Guidelines on Measurement Uncertainty* (CXG 54-2004)[4] and as adopted in the present guideline.

3.2.8 Lot size

Lot size is not normally an input required for the design of sampling plans intended to control both the consumer's and producer's risks in acceptance sampling. However, specification of the lot size is required for attributes plans applied to small lots and it is an input in the sampling plans described in the ISO 2859[2] and ISO 3951[3] standards (see sections 4.2.3, 4.3.4 and Appendix II).

[v] A network of organisations in Europe having the objective of establishing a system for the international traceability of chemical measurements and the promotion of good quality practices.

[vi] The Joint Committee for Guides in Metrology (JCGM).

[vii] See note v above.

4 Sampling plans

4.1 Selection of sampling plans

The following table provides direction to the relevant sections within these guidelines:

Table 2: Direction to the relevant part for the selection of sampling plans

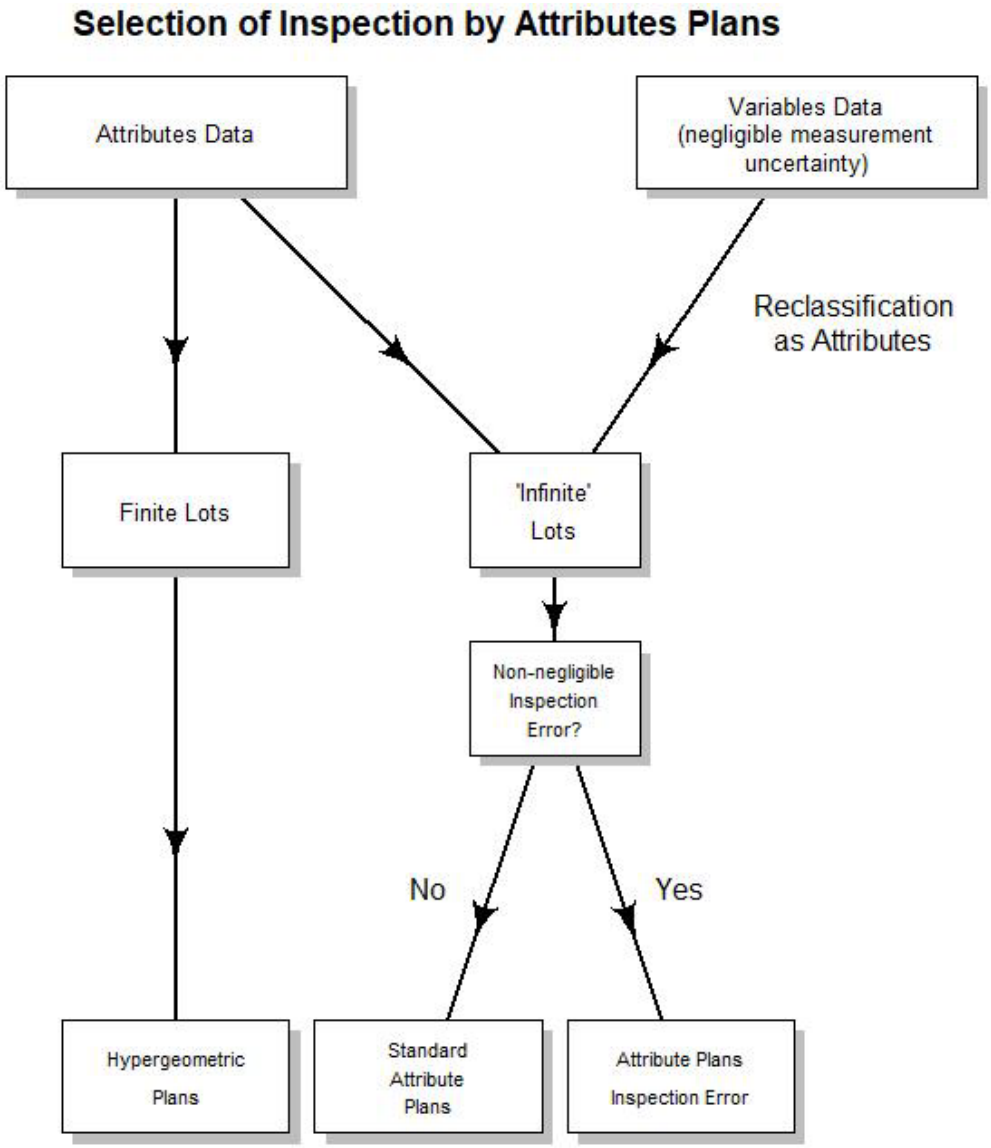
Homogeneous lots				
Data type	**Nature of provision**	**Distribution**	**Negligible** measurement uncertainty	**Non-negligible** measurement uncertainty
Attributes	Minimum or maximum	Not applicable	Inspection by attributes plans (section 4.2) Appendix II Table 8.4.1	Known inspection errors (section 5.1.1)
Variables	Minimum or maximum	Normal	Inspection by variables plans (section 4.3) Appendix II Table 8.4.2	Repeatability error (no laboratory bias) (section 5.2.6)
				General measurement uncertainty (sections 5.2.5, 5.2.7, 5.2.8)
				Fractional nonconformance plans (section 5.2.8)
	Minimum or maximum	Non-normal	Classification to attributes (Section 4.3.3)	Fractional nonconformance plans (section 5.2.8)
Variables	Minimum or maximum	Compositional proportions	Plans for compositional proportions (section 4.4.10)	Not included
	Average level	Not applicable	Plans for average level (section 4.3.5)	Not included
Inhomogeneous lots (bulk materials)				
Attributes	Minimum or maximum	(blank)	Attributes plans (section 4.4.6)	
Variables	Minimum or maximum	(blank)	Variables plans (section 4.4.9)	
	Average level	Not applicable	Plans for average level (section 4.4.8)	

4.2 Inspection by attributes plans

4.2.1 Introduction

These plans are usually referred to as attributes sampling plans. They are the simplest type of single sampling plan because the inspection results are classified into two possible outcomes – conforming or nonconforming. Because they are applicable to all sampling situations, they have become the benchmark that all other sampling plans can be compared against.

The following diagram shows the process for the selection of attributes sampling plans as it depends on the type of data and nature of the lot.



4.2.2 Two-class attributes plans

Two-class attributes plans are defined by two numbers: the sample size ***n***, the number of items to be taken from the lot under inspection and the acceptance number ***c***, the maximum number of nonconforming items allowed in the sample for acceptance of the lot. If the number of nonconforming items in the sample is less than or equal to ***c***, then the lot can be accepted. If the number of nonconforming items found is greater than ***c***, then the lot is rejected. In their most general form, the number of samples ***n*** and the acceptance number ***c*** for these plans are determined from specifications of the allowable consumer's and producer's risks. It should be noted that ***c*** need not be zero.

These plans can be used for either isolated lots or a continuing series of lots that consist of either discrete items or are bulk materials.

4.2.3 ISO standards – attributes plans

The ISO 2859[2] series of standards provides sampling plans that are indexed by either CRQ or PRQ. The lot size is an input to the sampling plans in these standards as the sample size depends on the lot size.

The ISO 2859-2[7] plans are indexed by CRQ and are intended for the inspection of isolated lots consisting of discrete items. These plans are suitable for application in the field of food safety when it is not appropriate to explicitly control producer risks in the design of the plans.

Appendix II contains tables for inspection by attributes plans from ISO 2859-1.[2]

These plans are indexed by the PRQ.

4.2.4 Plans for small lots (based on the hypergeometric distribution)

If the sample size is large in relation to the lot size, some economy in the number of samples may be possible. As a rule, such economies are possible if the number of items, calculated assuming an infinite lot size, exceeds 10 percent of the lot size. For conceptually infinite lots, sampling plans based on the hypergeometric distribution are the same as the general two-class plans based on the binomial distribution.

4.2.5 Zero-acceptance number plans

Zero-acceptance number (ZAN) plans are a special case of two-class plans in which the acceptance numbers are set to $\boldsymbol{c} = 0$. They are used in more critical situations such as for pathogens or foreign matter where only CR is considered directly and acceptance of lots demands that nonconforming items are not found in the inspection.

However, just because nonconforming items have not been found does not mean that they are not present in lots that have passed inspection. One disadvantage of ZAN plans is that they have poor discrimination between lots of good and poor quality, so they may not be generally applicable. The low sample numbers generally employed for microbiological applications enable high levels of consumer protection to be provided because of the offsets between the limits used in those plans and levels of contamination at which food might become unsafe (see section 3.2.4).

ZAN plans for finite lots can also be designed based on the hypergeometric distribution.

4.2.6 Three-class attribute plans

In these plans, inspection results are classified into three classes, usually referred to as ‘good’, ‘marginal’ and ‘poor’ or ‘unacceptable’. This type of plan is frequently used in microbiological assessments. They have an advantage, relative to two-class plans, of providing better discrimination between good and poor quality; they have ‘steeper’ OC curves than two-class plans for the same number of samples.

Three-class plans are defined by four numbers (***n, c, m, M***) where:

- ***n*** is the number of samples to be taken;
- ***c*** is the maximum number of ‘marginal’ samples allowed for acceptance of the lot;
- ***m*** is the limit separating good quality from marginal quality samples;
- ***M*** is the limit above which samples are classified as ‘poor’; and
- samples with results lying between the numbers ***m*** and ***M*** are classified as marginal.

Lots are accepted provided:

- none of the ***n*** samples is poor, having levels exceeding ***M***; and
- at most ***c*** of the samples are marginal, with levels between ***m*** and ***M***.

If $\boldsymbol{m} = \boldsymbol{M}$ a three-class plan becomes a two-class plan.

Evaluation of these plans generally requires an assumption about the underlying distribution of the identified characteristic, such as the lognormal distribution for microbiological parameters. This might also apply to two-class plans, especially for microbiological plans.

Three-class plans for finite lots can be designed based on the hypergeometric distribution.

4.2.7 Plans for variables data where an appropriate distribution is unknown

If the underlying distribution of a measured characteristic within a lot is not known and we are not prepared to assume that the characteristic can be adequately described by the normal or some other distribution, then the only recourse available is to classify the results as conforming or nonconforming with respect to the specification limit and to use attributes plans. Note that this approach should be used only when measurement uncertainty is negligible.

4.2.8 Attribute plans for multiple characteristics

Attributes plans can be easily applied to multiple characteristics by classifying inspected items as nonconforming if any of the individual characteristics are nonconforming.

Obviously, it makes sense to apply a plan to multiple characteristics only if the individual characteristics are of similar ‘stringency’, i.e. if the same or similar plans would be used if the characteristics were inspected individually. These plans have the advantage, compared to the use of individual plans, of allowing better control of PR, of incorrectly rejecting lots of good quality.

4.3 Inspection by variables plans

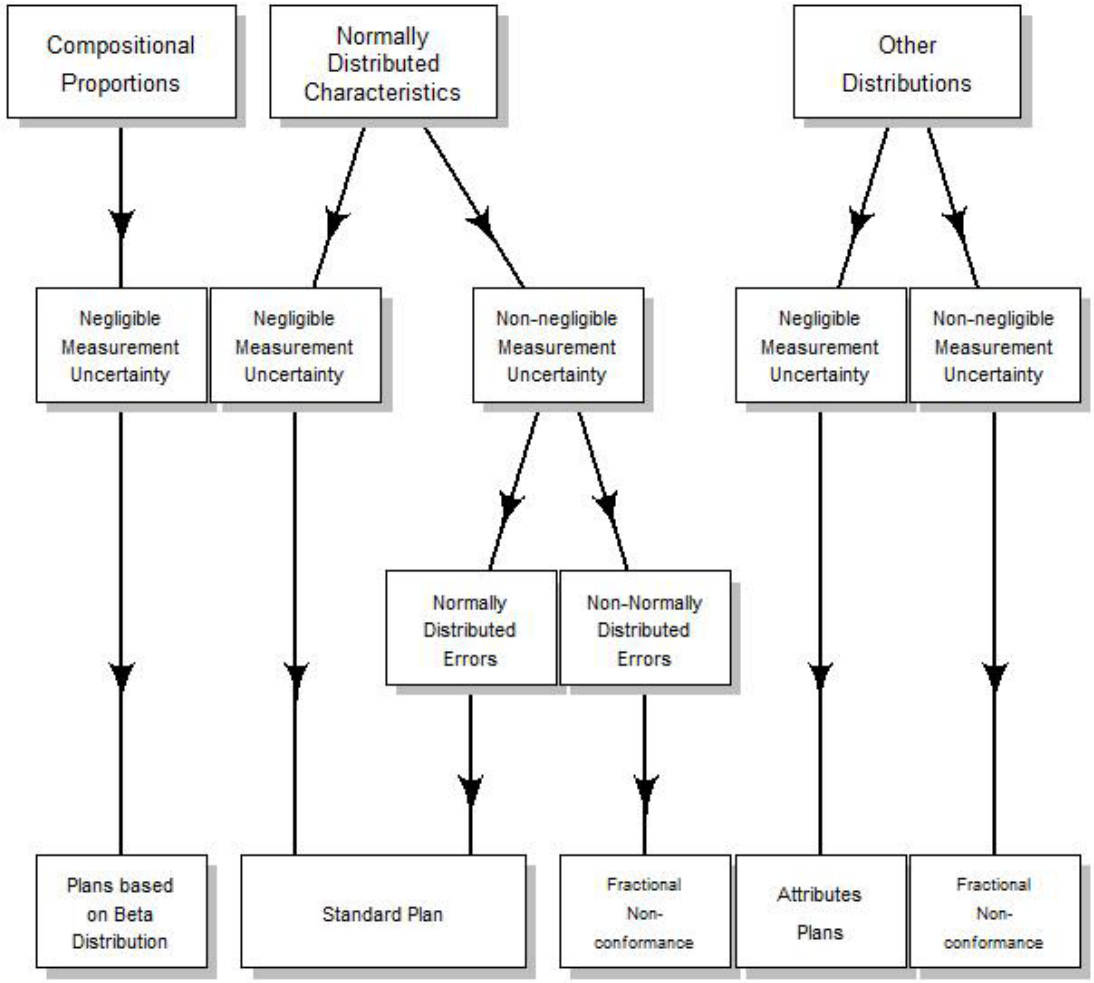
4.3.1 Introduction

If the underlying distribution of a measured characteristic is known, acceptance sampling can be performed directly on the measurements themselves. This often allows a considerable reduction in sample size.

For variables plans, it is necessary to make an assumption regarding the distribution of the characteristic within the lot. While the normal (Gaussian) distribution is commonly adopted, for compositional proportions in bulk materials the beta distribution is more appropriate (though the normal distribution can serve as an approximation).

The following diagram shows the process for the selection of variables sampling plans:

Selection of Inspection by Variables Plans - Homogeneous Characteristics



4.3.2 Advantages and disadvantages of variables plans

The advantages of variables sampling plans are:

- they offer the same protection with a smaller sample size than that required for attributes plans;
- there is feedback of data on the process which produced the units;
- there is more information available in waiver situations; and
- the extent of conformity of each unit is taken into account in the application of the plan.

The disadvantages are:

- the outcome is dependent on the appropriateness of the underlying distribution, that the assumed statistical distribution provides a satisfactory description for the behaviour of the characteristic within the lot;
- they are only applicable to one characteristic at a time;
- there may be a higher inspection cost per unit;
- a lot with no nonconforming units may be rejected by a variables plan, that occur when the average level lies too close to the specification limit, as measured in terms of the variation in the lot (lot standard deviation); and
- there is a possibility that no nonconforming units are found to show to the producer after rejection.

4.3.3 Variables plans

Variables sampling plans are defined by two numbers: the sample size n, the number of items to be taken from the lot under inspection, and the acceptability constant ***k***, the multiplier of the lot standard deviation ***S*** in the acceptance criterion.

A lot is accepted if $\bar{X} + kS \leq U$ for an upper specification limit U or if $\bar{X} - kS \geq L$ for a lower limit L.

4.3.4 ISO standards – variables plans

The ISO 3951[Error! Bookmark not defined.] standards provide sampling plans that are indexed by either CRQ or PRQ. The lot size is an input to the sampling plans in these standards as the sample size depends on the lot size.

The ISO plans indexed by CRQ are intended for the inspection of homogeneous isolated lots consisting of discrete items. These plans are more suited for provisions relating to food safety when it is not appropriate to explicitly control producer risks in the design of the plans.

Appendix II contains tables for inspection by variables plans from ISO 3951-1.[3] These plans are indexed by the PRQ.

The ISO 3951-6[8] standard also contains procedures that deal with non-negligible measurement uncertainty. This is discussed in more detail in section 5.

4.3.5 Plans for the average level in the lot

In some cases, such as the net weight of packages, a limit applies to the average level, with the intention that the average level in the lot should not be less than the limit. In Codex, although an example of sampling plans for bulk materials, the plans for aflatoxins are also based on compliance of the average level. This is an example of the use of offsets (see section 3.2.3).

It is usually assumed that the quality characteristic is normally distributed; the appropriateness of the distribution is less critical when compliance of the average level is being assessed. It is also usually assumed that there is a single specification limit, either a lower specification limit, ***L*** or an upper specification limit, ***U***.

When the lot standard deviation **σ** is known based on historical process data, the inspection plan for compliance of the average level to a minimum limit ***L*** is operated as follows:

1. take a random sample of size ***n*** and obtain the sample mean;
2. calculate $A = L + k \times \sigma$; and
3. if the sample mean $\bar{x} > A$ accept the lot; otherwise reject the lot.

The parameters of the plan are ***n*** and ***k***. Note that ***k*** does not denote the same quantity as in the usual variables plans. When the lot standard deviation **σ** is unknown, it is replaced with the sample standard deviation ***s***. The OC curve for this plan is less discriminatory than the plan when the standard deviation **σ** is known, and a greater sample size will be required to provide equivalent discrimination to that provided when the standard deviation is known.

4.4 Sampling of bulk materials

4.4.1 Introduction

Bulk materials are continuous, consisting for example of particles of different densities and sizes. It is impossible to consider a lot of a bulk material as a set of discrete items because there is no way of selecting the items in a way that is not biased when using simple random sampling.

Some general objectives of bulk sampling are:

- acceptance on a lot-to-lot basis;
- characterizing the material as to grade,[viii] any need for further processing, and its destination;
- determination of weight or content for purposes of payment;
- determination of properties that must be known so that the end use will be appropriate; and
- experimentation and analysis to determine further sampling procedures and uses of the material.

Sampling units are created at the time of sampling by means of some kind of sampling device. The sampling units change depending on different factors such as how the device is employed, and the conditions that the device is used under.

In bulk sampling, a lot is seen as being composed of mutually exclusive segments.

Sometimes the segments are obvious, such as when the material comes in boxes or bags.

Other times the segments are not obvious, and so they have to be artificially created. One way of doing this is by superimposing imaginary grids over the material.

4.4.2 Theory of sampling

The theory of sampling provides a comprehensive approach to the design of sampling procedures, the aim of which is to obtain a sample for laboratory analysis whose composition is an unbiased estimate of the average level of a lot. However, this sample would not, by itself, be useful for assessing conformance of a lot to minimum or maximum specification limits as an additional allowance is required to compensate for variation in the lot to enable such assessments to be made.

4.4.3 Terminology

The special nature of sampling for bulk materials has led to the use of specific terminology, although this terminology varies between different fields, between authors, and also between different Codex committees. The *General Standard for Contaminants and Toxins in Food and Feed* (CXS 193-1995)[9] uses the following terminology.

Table 3: Bulk material terminology for sampling plans

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	It is defined by a test procedure and an accept/reject limit. A test procedure consists of three steps: sample selection, sample preparation and quantification. The accept/reject limit is a tolerance usually equal to the Codex maximum level.
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.
Laboratory sample	The smallest quantity of a food commodity comminuted in a mill or homogenized in an appropriate device. The laboratory sample may be a portion of or the entire aggregate sample. If the aggregate sample is larger than the laboratory sample, the laboratory sample should be removed in a random manner from the aggregate sample in such a way to ensure the laboratory sample is still representative of the sublot sampled.
Test portion	A portion of the comminuted/homogenized laboratory sample. The entire laboratory sample should be comminuted in a mill or homogenized in an appropriate device. A portion of the comminuted/homogenized laboratory sample is randomly removed for analysis.

[viii] Foods and other materials are often ranked according to their quality, with the different quality levels are sometimes known as grades.

4.4.4 Design of general sampling plans for bulk materials

In the simplest case, such as the inspection of bulk materials of manufactured products, lots can often be considered homogeneous, allowing the standard attributes or variables plans to be used, with adjustment for analytical measurement uncertainty where appropriate.

On the other hand, some bulk materials, such as shipments of grains or other raw materials, cannot be considered homogeneous (see section 3.2.7). Special techniques are required for this situation, but the statistical methods are complex and only an overview is provided in these guidelines.

Lot homogeneity is difficult to verify for bulk materials and generally requires large numbers of samples. Moreover, it is often difficult to perform random sampling from an entire lot of a bulk material. As a precaution, in cases where lot homogeneity can be neither assumed nor verified, lots should be treated as inhomogeneous.

The general approach to sampling inhomogeneous lots of bulk materials is that a lot is considered as a set of smaller segments (strata) each of which is more homogeneous than the entire lot. This allows the usual sampling procedures based on random sampling to be applied within each segment as inhomogeneity within each segment will have less effect.

The basic sampling and inspection procedure can be described as follows:

- segments, from which increments are to be taken are chosen at random;
- several increments are chosen at random from each of the chosen segments;
- the increments from each segment can sometime be combined to form a composite sample, which is thoroughly mixed;
- one or more subsamples are taken from each composite sample;
- these subsamples are tested; and
- acceptability of the lot is decided based on an acceptance criterion.

4.4.5 Attributes plans for bulk materials

The following points need to be considered in the design of attributes plans for bulk materials:

- inhomogeneity will be present and hence the standard attribute sampling plans for homogeneous lots will not be suitable as they do not provide adequate protection for consumers;
- inhomogeneity can be overcome either by allowing for the correlation within the batch in the design of the sampling plan or, alternatively, by splitting the lot into more homogeneous segments, and using stratified sampling techniques. Either way, a preliminary study is needed to estimate the correlation and the variation between segments; and
- the proposed plans should be validated using different statistical models for the behaviour of the level nonconforming within the lot, to ensure robustness against different levels of correlation.

4.4.6 Variables plans for bulk materials

Typically, the total observed variation within a lot of bulk materials consists of several components due to variation between and within segments, due to sample preparation (e.g. including subsampling), testing and other causes.

Sampling plans for bulk materials, especially cost-optimal sampling plans, can be designed most effectively with prior knowledge of the different components of variation that exist within lots; it is desirable that a preliminary investigation of the variation is carried out prior to the development of any plans.

A minimum of ten samples per segment is recommended to estimate the within lot variability, if the acceptance criterion involves averaging of multiple test results, laboratory samples should be tested at least in duplicate to allow estimation of the repeatability component of measurement uncertainty, unless an estimate is available from other sources such as a method validation study.

Example
The *General Standard for Contaminants and Toxins in Food and Feed* (CXS 193-1995)[9] shows the breakdown of the total variation for aflatoxins in tree-nuts, with a focus on sampling, sample preparation and testing; the variation due to sampling includes both between and within segment variation. It should be noted that provisions for aflatoxins are expressed in terms of the average levels in a lot.

Table 1. Variances[a] associated with the aflatoxin test procedure for each treenut

Test procedure	Almonds	Hazelnuts	Pistachios	Shelled Brazil nuts
Sampling[b,c]	$S^2_s = (7\,730/ns)\ 5.759C^{1.561}$	$S^2_s = (10\,000/ns)\ 4.291C^{1.609}$	$S^2_s = 8\,000/ns)\ 7.913C^{1.475}$	$s_s^2 = (1\,850/ns)\ 4.8616C^{1.889}$
Sample Prep[d]	$S^2_{sp} = (100/nss)\ 0.170C^{1.646}$	$S^2_{sp} = (50/nss)\ 0.021C^{1.545}$	$S^2_{sp} = (25/nss)\ 2.334C^{1.522}$	$s_{ss}^2 = (50/nss)\ 0.0306C^{0.632}$
Analytical[e]	$S^2_a = (1/na)\ 0.0484C^{2.0}$	$S^2_a = (1/na)\ 0.0484C^{2.0}$	$S^2_a = (1/na)\ 0.0484C^{2.0}$	experimental $s_a^2 = (1/n)\ 0.0164C^{1.117}$ or FAPAS $s_a^2 = (1/n)\ 0.0484C^{2.0}$
Total variance	$S^2_s + S^2_{sp} + S^2_a$	$S^2_s + S^2_{sp} + S^2_a$	$S^2_s + S^2_{sp} + S^2_a$	$S^2_s + S^2_{sp} + S^2_a$

S_s^2, S_{sp}^2 and S_a^2, denote the variance associated with the sampling, sample preparation and analytical steps, respectively.

A sampling plan is defined in terms of laboratory sample size *ns*, test portion size *nss* and the number of aliquots *na* (i.e. the number of analytical samples taken from each subsample). The information in this table can be used to design an optimal sampling plan in terms of total cost for a specified consumer's risk at a given concentration *C*. Obviously, the costs associated with each step need to be known to derive a cost-optimal plan.

Since bulk materials are continuous, parts of each sample can be mixed to form a composite sample. This composite is then tested only once, rather than having to perform many tests on the individual samples. This is a physical way of creating a sample representing the average content per lot or segment. This averaging causes a reduction in the apparent variation meaning that adjustment of the acceptance criterion may be required for assessments against minimum or maximum limits.

Note however, that the use of composite sampling adds complexity to the design of a general sampling strategy due to the statistical complexity of modelling the mixing process; assuming that composites made up from many individual portions can be thoroughly mixed is possibly unrealistic.

4.4.7 Variables plans for the average level

Sampling plans for bulk materials are often used to assess compliance of the average level of a characteristic. In some cases, such as in the sampling plans for aflatoxins in the *General Standard for Contaminants and Toxins in Food and Feed* (CXS 193-1995),[9] these plans are used in conjunction with offsets (see section 3.2.3) to provide consumer protection.

Other procedures for the inspection of the average level of a lot such as those in ISO 10725[10] are available that consider costs to derive plans that are economical to apply, although these plans might not be suitable in cases where a more precise determination of the average level is required.

Plans for the average level might also be applicable where the product is homogenized through blending or further processing.

4.4.8 Variables plans for percentage nonconforming (minimum or maximum limits)

The strategy is similar to the design of variables plans for the average level except that an additional allowance should be made for variation within the lot, obtainable from the statistical analysis described in section 4.4.5. A simpler approach is to estimate within lot variation as the variation among the segments by taking one sample from each segment and testing those samples in duplicate to allow adjustment for measurement uncertainty, although this will not provide any information on other components of variation:

- the acceptance criterion has the same form as a conventional variables plan applied to homogeneous lots; and
- the number of samples ***n*** and the acceptability constant ***k*** can be found by trial and error, assessing the probabilities of acceptance against various alternative models for the behaviour of the characteristic in the lot. This should recognize that the formation of the segments might not reflect the disposition of nonconforming product within the lot.

4.4.9 Variables plans for compositional proportions (measurement uncertainty negligible)

Compositional characteristics are often quality measures for bulk materials. For example, the milkfat percentage with a minimum limit of 26 percent is a primary quality measure for whole milk powders.[ix]

Compositional proportions, also referred to as mass fractions, are characterized by units of measure such as percent (of mass), mg/kg, µg/100g and the like, which are, strictly speaking, 'dimensionless' numbers lying between 0 and 1.

Compositional proportions can be modelled using the beta distribution. Variables sampling plans based on the normal distribution can only be approximate for compositional proportions and can lead to a higher CR than desired.

Sampling plans for compositional proportions are defined by two parameters, ***m***, the number of samples to be taken from the lot and ***k***, the acceptability constant defined in the same way as for the usual variables sampling plans. In order to design such plans, in addition to PRQ, CRQ etc., an estimate of the 'precision parameter' for the beta distribution, denoted by ***θ***, is required. This estimate can be obtained from the analysis of historical data.

When using these plans, the ***m*** samples are taken from the lot and can be tested individually or combined (blended, well mixed etc.) to form a composite sample that needs to be tested only once.

The average level ***P*** is taken as either the average of the ***m*** results from the testing of the individual samples or the single result from the testing of the composite sample.

A feature of the beta distribution is that its standard deviation depends on the average level, enabling an assessment to be conducted using a single test of a composite sample taken from the lot. The standard deviation is calculated using the formula:

$$s = \sqrt{P(1-P)/\theta}$$

where θ is the precision parameter for the beta distribution, estimated from historical data (see above).

The lot is accepted against an upper limit U provided $P + k \times s \leq U$ and similarly for a lower limit.

5 Inspection error and measurement uncertainty

Inspection error relates to inspection by attributes, and measurement uncertainty relates to inspection by variables.

Non-negligible analytical measurement uncertainty and inspection error have the potential to affect the probabilities of acceptance of a sampling plan. Accordingly, non-negligible analytical measurement uncertainty or inspection error should be taken into account in sampling inspection.

It has been shown theoretically that analytical measurement uncertainty and inspection errors affect the PR more than they affect the CR, i.e. the increase in PR (rejecting a lot of acceptable quality) exceeds the increase in CR (accepting a lot of unacceptable quality). Accordingly, in the interests of fairness, it is important that appropriate allowances are made for non-negligible measurement and inspection errors.

Acceptance sampling plans can be designed to allow for non-negligible analytical measurement uncertainty and inspection error.

5.1 Attributes plans

In the context of attributes plans, 'inspection error' refers to random errors of misclassifying conforming items as nonconforming and vice versa.

Inspection errors occur when testing an item for conformance and can be caused by human error, instrument error, or any other measurement-related errors.

There are two types of inspection errors:

[ix] *Standard for Milk Powders and Cream Powders* (CXS 207-1999)

- Type I errors (e_1) occur when conforming items are classified as nonconforming.
- Type II errors (e_2) are when nonconforming items are classified as conforming.

When inspection errors are present, they generally cause a greater increase in producer's risk than CR. For a single sampling plan, Type I errors (e_1) have a greater effect on the OC curve than Type II errors (e_2).

The true fraction nonconforming p and the observed fraction nonconforming p_e are related through the following equation:

$$p_e = e_1(1-p) + (1-e_2)p$$

The impact of inspection errors is particularly marked for ZAN plans.

5.1.1 Known inspection errors

If the misclassification errors are known, if precise estimates of the misclassification errors are available, for example from a method validation study, the estimates of the Type I and Type II errors can be used to design a sampling plan to control producer's and consumer's risks to specified levels. This will inevitably lead to increased sample sizes.

5.2 Variables plans

Measurement uncertainty provides information regarding the range of values that could reasonably be attributed to the measurand. As such, it constitutes an important measure of the quality or reliability of a test result.

For a more comprehensive discussion of measurement uncertainty, refer to the *Guidelines on Measurement Uncertainty* (CXG 54-2004).**Error! Bookmark not defined.**

It should be noted that the concept of measurement uncertainty as usually understood (and as discussed in the *Guidelines on Measurement Uncertainty* [CXG 54-2004])**Error! Bookmark not defined.** relates to a single determination performed on a single sample. This is appropriate for conformity assessment, but not for acceptance sampling (see section 2.2). The same holds for the procedure illustrated in Figure 1 in the *Guidelines on Measurement Uncertainty* (CXG 54-2004).**Error! Bookmark not defined.** In connection with acceptance sampling, it is important to take into account how the different measurement uncertainty components manifest themselves in the sampling and calculation procedures applied. This is discussed in section 5.2.4, below.

The terms 'negligible' and 'non-negligible'[x] are used to indicate whether or not allowances should be made for measurement uncertainty in acceptance sampling plans. In the ISO 3951**Error! Bookmark not defined.** series, measurement uncertainty is considered non-negligible if it is greater than 10 percent of the *process* standard deviation (SD). In connection with the inspection of isolated lots, the same criterion can be applied, but replacing the *process* SD with the *lot* SD (see section 3.2.6). However, the only definitive way to assess whether an adjustment for measurement uncertainty is required is to examine the OC curve for the proposed sampling plan in the presence of measurement uncertainty (see section 2.3.1).

5.2.1 Measurement uncertainty

In order to clarify the role of measurement uncertainty in acceptance sampling, it is necessary to draw a distinction between *analytical* measurement uncertainty and the *sampling component* of (the total) measurement uncertainty. We start by reproducing the following definition from section 8 in the *Guidelines on Measurement Uncertainty* (CXG 54-2004):**Error! Bookmark not defined.**

A laboratory sample is a sample as prepared (from the lot) for sending to the laboratory and intended for inspection or testing.

Any sources which contribute to measurement uncertainty prior to the arrival of the laboratory sample in the laboratory can be considered components of sampling uncertainty:

- the sampling procedure and its implementation;
- the variation of the characteristic of interest within the lot;
- the person(s) performing the sampling;
- subsampling steps (leading to the laboratory sample); and
- contributions due to storage and transportation conditions (prior to the arrival of the laboratory sample in the laboratory).

[x] The term 'significant' is also used.

Any sources which contribute to uncertainty within the laboratory can be considered components of analytical measurement uncertainty, for example:

- subsampling steps performed on the basis of the laboratory sample, such as taking a test sample, test portion, etc.;
- sample preparation;
- contributions due to storage conditions (in the laboratory);
- analytical steps; and
- laboratory procedures.

In determining measurement uncertainty, it is important to take account of all relevant contributions, including all sampling and analytical sources.

Role of measurement uncertainty in acceptance sampling

In acceptance sampling, the aim is to decide whether to accept or reject the lot under inspection via the application of an acceptance criterion. The application of the acceptance criterion often includes an estimate of the lot SD, which is a measure of the random variation of the characteristic within the lot under inspection. It is important to ensure the estimate of the lot SD is not affected by uncertainty sources. Accordingly, the role of measurement uncertainty in acceptance sampling can be described as follows:

> *Measurement uncertainty may affect the estimate of the lot SD. If this effect is non-negligible and thus impacts the consumer and producer risks, then the estimate of the lot SD must be corrected for the non-negligible measurement uncertainty.*

In theory, the estimate of the lot SD can be affected by both sampling and analytical components of measurement uncertainty. It should be noted, however, that while analytical uncertainty will always inflate the lot SD estimate, the effect of sampling components can be either to increase or decrease its value. For this reason, correcting the estimate of the lot SD for analytical uncertainty will always consist in 'subtracting' the uncertainty contribution and can thus be considered more readily achievable than a correction for sampling uncertainty components. The focus in this guidance document thus lies on correcting for non-negligible *analytical* uncertainty. Notwithstanding, it should be ensured sampling procedures are adequate. The use of statistically-based random sampling or validated sampling procedures is desirable. It should also be noted that any impact of analytical or sampling uncertainty on the lot SD estimate can be disregarded as long as the corresponding SD is less than 10 percent of the lot SD.

Procedures for correcting the lot SD for non-negligible analytical measurement uncertainty and sampling uncertainty are discussed in the following sections (see section 5.2.6).

5.2.2 General discussion of bias

Measurement uncertainty consists, on the one hand, of components that reflect random effects (varying randomly with each test result) and, on the other hand, of components that reflect systematic effects (remaining constant across test results).

A systematic effect is commonly referred to as a bias.

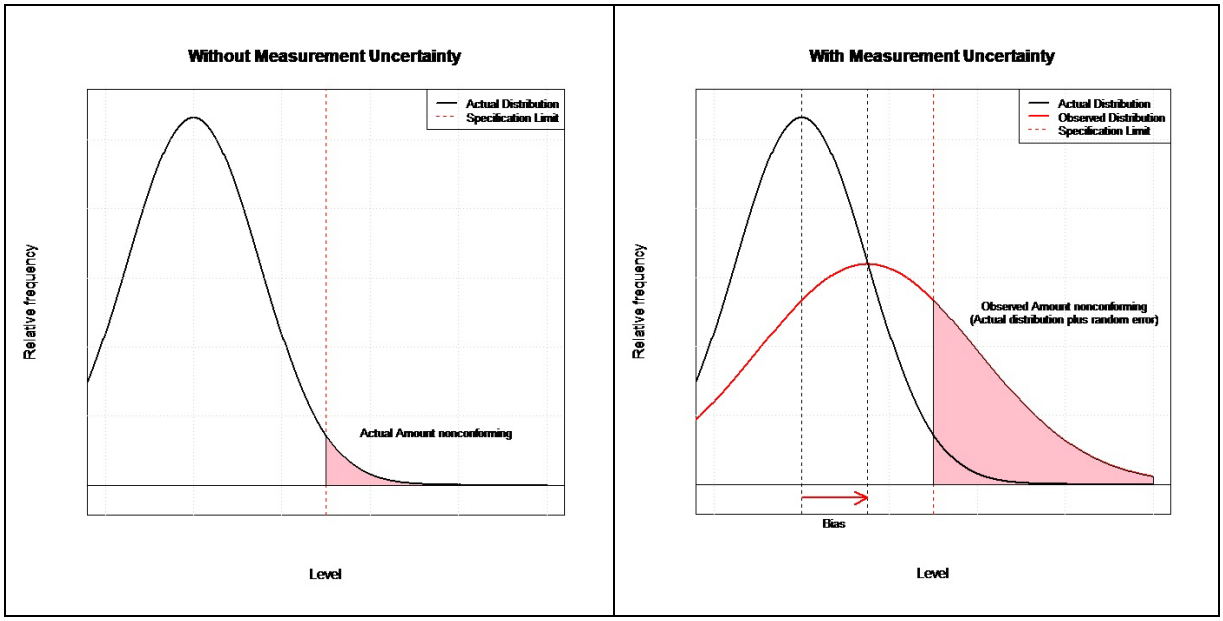
In principle, if a bias is observed, it is corrected for; and it is the *uncertainty of the bias correction* which is taken into account in the measurement uncertainty.

In practice, a bias may affect test results even after a *bias correction* is performed. This is the case, for example, if the bias correction is adequate for a given matrix, but not for another.

There may be various sources of bias. The analytical method itself may have a bias. In addition, the method bias may vary from one matrix to the next. In this sense, matrix effects (or a 'matrix bias') may be observed. The method bias may vary from one laboratory to the next. In this sense, laboratory effects (or a 'laboratory bias') may be observed. Finally, there may also be a sampling bias, e.g. a given sampling procedure may consistently underestimate the lot mean or the lot SD.

It is often possible to obtain an estimate of the magnitude of a bias even in the absence of information regarding the 'true value'. For instance, the 'between-laboratory' component of reproducibility precision, calculated on the basis of data from a collaborative study, and typically expressed as a SD, characterizes the magnitude of the laboratory bias. Similarly, there are procedures for estimating laboratory bias on the basis of quality control data or proficiency test results which can be used to characterize the magnitude of the laboratory bias.

The following diagram shows the distribution and the percent nonconforming in a lot in the case that there are neither random effects nor bias (seered to as an 'error-free' plan), and the effect which random effects and bias can have on the observed distribution and the apparent percentage nonconforming in a lot. This diagram thus shows the effect that random effects and bias can have on the probability of acceptance of a lot, unless such effects are adequately accounted for.



5.2.3 Top-down approach for determining measurement uncertainty: the ISO 5725-2[11] model

In many cases, an estimate of analytical measurement uncertainty is supported by precision data from an inter-laboratory method validation study (collaborative study) calculated on the basis of the simple design from the ISO 5725-1[12] and ISO 5725-2[11] standards. This design allows two precision components to be calculated:

- one component reflecting *random effects* under near identical conditions within a given laboratory, referred to as the repeatability component; and
- one component reflecting *laboratory bias*, referred to as the between-laboratory component.

The underlying statistical model is not the most general model,[xi] but many collaborative studies are conducted in accordance with ISO 5725-2.[11] For this reason, the following sections will return to the two components of the ISO 5725-2 design.

Note:The 'between-laboratory' component in ISO 5725-2[11] characterizes the range of laboratory bias under repeatability conditions. ISO 5725-3 [13] includes other designs, which allow a separate estimation of repeatability precision, intermediate precision (factorial effects) and residual laboratory bias.

5.2.4 The acceptance criterion

The acceptance criterion in a variables plan often takes the form:

$$\bar{x} + k \cdot s \leq USL,$$

where $\bar{x}$ is the average value of the test results obtained from the inspection, s is their standard deviation and USL denotes the upper specification limit.

Ideally, the standard deviation s is a reliable measure of the variation of the characteristic of interest within the lot. However, in practice, s may include other components, such as, analytical measurement uncertainty.

- The mean value $\bar{x}$ is calculated from several test results. When taking measurement uncertainty into account in the acceptance criterion, it is thus necessary to consider how averaging affects the different components of analytical measurement uncertainty.

As far as the two components from the ISO 5725-2[11] model discussed above:

- averaging across n test results will reduce the repeatability component by a factor of $\mathrm{sqrt(n)}$; and
- however, averaging across n test results will not reduce the between-laboratory component.

[xi] For common top-down approaches, see the Guidelines on Measurement Uncertainty (CXG 54-20004).

In the absence of fundamental variability, the lot standard deviation from a *single* test result obtained from a *well-mixed* composite sample obtained from n increments is reduced by $\mathrm{sqrt(n)}$.

5.2.5 Laboratory bias in acceptance sampling

In connection with acceptance sampling, the following should be noted:

- If information regarding laboratory bias is available in the form of a between-laboratory SD from an interlaboratory study conducted according to ISO 5725-2,[11] then measurements during lot inspection should be performed under repeatability conditions, with the bias, represented by the between-laboratory SD, taken into account in the sampling plan.
- Matrix effects (variation of bias across matrices within the scope of the method) can affect the test results differently in different laboratories (see the *Guidelines on Measurement Uncertainty* [CXG 54-2004]),[Error! Bookmark not defined.] sections 10, 12 and 15. This means that an estimate of the between-laboratory variation may be valid for a given matrix, but not for another. An estimate of the bias across different matrices can be obtained by means of an in-house experiment. If such an estimate is available, it should be taken into account in the sampling plan.

If an estimate of the between-laboratory SD is available, it is important to consider whether it constitutes a reliable characterization of the variation of laboratory bias, in the sense that the estimate was obtained on the basis of data from a sufficiently large number of laboratories (see the *Guidelines on Measurement Uncertainty* [CXG 54-2004],[Error! Bookmark not defined.] sections 16, 17 and 18).

5.2.6 Within-item variation

For the case of lots consisting of discrete items, one uncertainty source deserves special attention: **within-item variation**. Typically, one measurement value is obtained per item, and the lot SD is calculated on the basis of these item-specific values. Each measurement value is intended to represent the mean concentration of the given item. However, the lot SD calculated in this manner may be inflated by within-item variation. There are two cases to consider.

Case 1 – subsampling prior to the arrival of the sample in the laboratory

In this scenario, there is a subsampling step between item selection and the arrival of the laboratory sample in the laboratory, and this subsampling step causes non-negligible deviations between laboratory samples from one and the same item (if several laboratory samples were taken from the same item). Note that in this case, the lot SD will be inflated by a sampling (rather than an analytical) component of measurement uncertainty. Correcting for this type of overestimation of the lot SD presents practicability issues and is not typically contemplated. This case is mentioned here merely for the sake of completeness.

Case 2 – subsampling within the laboratory

In this scenario, subsampling inside the laboratory causes non-negligible deviations between test portions taken from the same laboratory sample (item). Conceptually, this component belongs to analytical rather than sampling measurement uncertainty. An estimate thereof can be obtained via a 'duplicate' experimental design, where two test portions per laboratory sample (item) are analysed. If a validation study is conducted on the basis of certified reference material, it may not be possible to obtain an estimate of this component. Moreover, depending on the context, this component may or may not be considered to belong to a given method's precision. Accordingly, in some cases, an estimate for this component may not be available at all, or may only be available via studies conducted to determine sampling uncertainty rather than analytical uncertainty.

5.2.7 Absence of laboratory bias

In order to ensure unbiased estimates, the estimate of the lot SD must be corrected for any unwanted measurement uncertainty and subsampling components (as described under Case 2 in the previous section). In the absence of laboratory bias, it is possible achieve this via a relatively simple procedure.

If it can be assumed that:

- there is negligible bias;
- the characteristic follows a normal distribution in the lot under inspection; and
- repeatability effects follow a normal distribution;

then the following approach can be applied.

The standard deviation s is adjusted by 'subtracting' the standard deviation representing the repeatability component of measurement uncertainty u:

$s_{adj}^2 = s^2 - u^2$. The adjusted SD is then used in the acceptance criterion:

$\bar{x} + ks_{adj} \leq USL$. If the measurement uncertainty is greater than s, the adjusted standard deviation is set equal to zero.

If there is no subsampling variation, then the procedure described above is adequate.

If the lot SD is inflated by a subsampling component and u reflects this component, then the procedure described above is adequate.

If the lot SD is inflated by a subsampling component (as described under Case 2 in the previous section), and if u *does not* reflect this component, then another approach can be used to adjust the lot SD for both repeatability and the between-subsample variation. In particular, if every item is tested in duplicate, an adjustment for measurement uncertainty can be made for both subsampling variation and repeatability. In this case the observed standard deviation *s* calculated from all the data is adjusted by subtracting the quantity $½u^2$ where *u* is the standard deviation of the differences between the results for each pair of duplicate samples:

$$s_{adj}^2 = s^2 - ½u^2.$$

5.2.8 Presence of laboratory bias

We consider the case that that an estimate of between-laboratory variation is available, e.g. from a validation study previously conducted in accordance with ISO 5725.[12]

This estimate is considered a measure of laboratory bias and is taken into account in the sampling plan.

If the laboratory bias is relatively small, allowance can be made using the techniques described in Annex B of ISO 3951-6.[8] It is assumed that repeatability and laboratory-bias effects, as well as the characteristic, are normally distributed. While the acceptance criterion is of the same form as in the 'error-free' variables plans, in some circumstances it might not be possible to find a sampling plan (the number of samples ***n*** and the acceptability constant ***k***) that controls producer's and consumer's risks in the manner intended.

If the laboratory bias (i.e. the estimate of between-laboratory variation) is too large to apply the procedure from ISO 3951-6,[8] then an adjusted specification limit USL_{adj} should be calculated as $USL_{adj} = USL - q \cdot s_L$,

where s_L denotes the estimate of between-laboratory variation (expressed as a standard deviation) and q denotes the appropriate quantile. If an estimate of the variation of bias across matrices s_{matrix} is available, then the adjusted specification limit should be calculated as:

$$USL_{adj} = USL - q \cdot \sqrt{{s_L}^2 + {s_{matrix}}^2}.$$

5.2.9 Fractional nonconformance

If the characteristic does not follow a normal distribution (see section 3.2.5), plans based on fractional nonconformance (FNC) can be used to allow for analytical measurement uncertainty.

The FNC for a sample can be thought of as the probability that the true value of the sample exceeds the specification limit, allowing for any measurement uncertainty present.

A sampling plan based on the FNC adjustment principle is defined by two numbers, ***n***, the number of samples to be taken and ***Ac***, the maximum acceptance limit for acceptance of the lot. These two numbers are determined in the same manner as for other types of plans, namely, by considering the allowable risks at PRQ and CRQ. Additional information on the ratio between measurement uncertainty and lot SD is also required for the design of these plans.

A lot is accepted provided the sum of the individual sample FNC values does not exceed the maximum acceptance limit.

$$\sum_{i=1}^{n} FNC_i \leq Ac$$

where FNC_i is the FNC value for the i^{th} sample ($i = 1 \ldots n$).

The use of FNC adjustment is preferred over approaches in which samples are classified as conforming or non-conforming against a specification limit or on a 'beyond reasonable doubt' basis taking measurement uncertainty in account. Such approaches are less economical in terms of sample numbers and might not be optimal in terms of controlling producer's and consumer's risks and need to be evaluated.

6 Other matters relating to sampling

6.1 Physical sampling

The theory of sampling (see section 4.4.2) relies on procedures that represent best practice for unbiased physical sampling from a lot. These sampling procedures should be observed with respect to each individual sample taken from a lot, and for any subsequent mixing and subsampling etc., noting that usually more than a single sample is required in acceptance sampling plans. Reference should be made to material-specific ISO or other standards for details of sampling procedures for different commodities. Adherence to specified sampling procedures might be a legislative or regulatory requirement for some commodities in some jurisdictions.

6.1.1 Random sampling

For lots consisting of discrete items, random sampling means that each item has an equal chance of being selected in the sample. The assumption of random sampling allows the operating characteristic to be calculated; deviating from random sampling might mean that the plan does not control the producer's or consumer's risks as might have been intended. In many cases systematic sampling, taking samples at regularly spaced intervals throughout a lot, will suffice as a substitute for true random sampling.

It is common for lots to be 'layered', individual items might be packed in cartons, there might be several (but the same number) of these smaller cartons packed into a larger carton, and several (but the same number) of the larger cartons packed on a pallet. Selecting a random sample of size ***n*** items would proceed as follows:

- select ***n*** pallets from the number of pallets in the lot (the same pallet can be selected more than once);
- select a random larger carton from the cartons on each side of the selected pallets;
- select a smaller carton from each of the larger cartons that have been selected; and
- finally, select an individual item from each of these smaller cartons – these constitute the sample which will be tested or examined.

For bulk materials taking a random sample is more difficult. Many lots of bulk materials can be considered as a collection of segments; stratified random sampling is used in which, in the simplest case, segments are selected at random from the total number of segments, then within each segment that has been chosen a random sample of increments is taken.

This is discussed in more detail in section 4.4.

In principle, there is no need for random sampling for well-mixed fluids or bulk products; however random sampling might still be used as a precaution against inhomogeneity or for procedural reasons.

6.1.2 Convenience sampling

Convenience sampling is often referred to as pragmatic sampling. It involves taking samples, and sometimes only a single sample, from a part of a population that is convenient to sample and is often used due to low cost. It is a form of ad hoc sampling that is sometimes used in pilot testing.

There are usually more disadvantages than advantages with convenience sampling. There is a possibility of sampling error and lack of adequate representation of the population, and furthermore, use of convenience sampling might lead to disputes as it is neither a fair nor a valid procedure.

6.2 Inhomogeneous lots

While section 3.2.4 discusses the *conditions under which* a lot can be considered homogeneous, this section addresses the question *how to handle* cases of inhomogeneous lots consisting of discrete items. For more information on sampling of inhomogeneous lots consisting of bulk materials, refer to section 4.4.

Most sampling plans are based on the assumption that the lots are homogeneous. Use of these plans with inhomogeneous lots will usually increase producer's risks and consumer's risks, so that consumer protection may be compromised.

Lots may be inhomogeneous because inspection lots differ from manufacturing lots. Accordingly, one approach may be to split a given inhomogeneous inspection lot into sublots in line with production lots or other standardized manufacturing processes. Each of the sublots might then be sufficiently homogeneous to be inspected using standard attributes or variables sampling plans, inspecting each sublot with the same plan that would have been used for the entire lot, had it been homogeneous. However, lots should not be split into sublots based on results obtained from earlier testing.

APPENDIX I

GUIDE TO THE SELECTION AND DESIGN OF SAMPLING PLANS

1 Introduction

The concepts and criteria for sampling plans described in these guidelines are applicable to provisions in Codex standards. This Appendix provides a guide to the design of those sampling plans.

It has been structured in a way that allows users to follow the process for the design of a sampling plan from first principles to quickly identify options for sampling plans that are relevant to a particular situation in which sampling is to be undertaken.

Links are provided that allow users to quickly access further information about particular sampling options in the main document.

1.1 Starting point

The following examples are provided to assist in the design of sampling plans and should not be understood as a recommendation.

Example: Options for attributes sampling plans

In the following, the producer's risk (PR) is 5 percent and the CR is 10 percent. These values are commonly used.

Attribute sampling plans with producer's risk quality (PRQ), the quality level at which the lot of 6.5 percent may apply to commodity defects such as blemishes and other visual defects on fresh fruit.

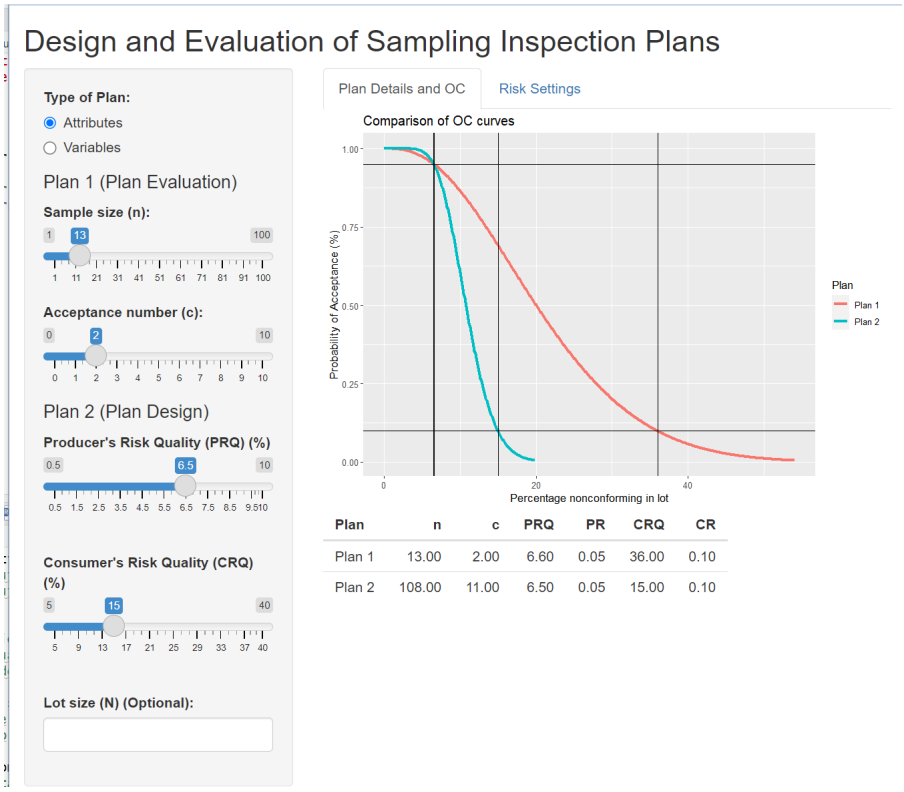
The PRQ of 6.5 percent means that lots containing 6.5 percent of nonconforming items will be accepted 95 percent of the time whereas, for example, a consumer's risk quality (CRQ) of 20 percent means that lots containing 20 percent of nonconforming items will be rejected 90 percent of the time.

The following table shows options for sampling plans for different levels of CR quality.

Table: Sampling plan options for PRQ = 6.5 percent

CRQ	PRQ	n	c
20%	6.5%	51	6
25%	6.5%	30	4
30%	6.5%	21	3
36%	6.5%	13	2

The operating characteristics for two of these plans is shown below; this shows the probability of accepting a lot with those plans at any quality level. The choice of a sampling plan will depend on the probability of acceptance across the entire range of quality levels.



Plan	n	c	PRQ	PR	CRQ	CR
Plan 1	13.00	2.00	6.60	0.05	36.00	0.10
Plan 2	108.00	11.00	6.50	0.05	15.00	0.10

Example: Options for variables sampling plans

The provison for a compositional characteristic for a commodity specifies that the percentage content should not exceed a maximum limit. In this example it is assumed that the measurement uncertainty is negligible and that the lot SD is known.

The following table shows options for variables sampling plans with a PRQ of 3.5 percent and for different levels of CR quality.

Table: Sampling plan options for PRQ = 3.5 percent

CRQ	PRQ	n	k
10%	3.5%	31	1.52
15%	3.5%	16	1.39
20%	3.5%	10	1.29
25%	3.5%	7	1.19
30%	3.5%	6	1.14
35%	3.5%	5	1.08

The operating characteristics for two of these plans is shown below; this shows the probability of accepting a lot with those plans at any quality level. The choice of a sampling plan will depend on the probability of acceptance across the entire range of quality levels. It will also depend on whether the lot SD is known or unknown.

Design and Evaluation of Sampling Inspection Plans

Type of Plan:

- ○ Attributes
- ◉ Variables

Standard Deviation Type

- ◉ Known
- ○ Unknown

Plan 1 (Plan Evaluation)

Sample size (n):

1 — 28 — 100

1 11 21 31 41 51 61 71 81 91 100

k-constant (k):

1 — 1.38 — 3

1 1.2 1.4 1.6 1.8 2 2.2 2.4 2.6 2.8 3

Plan 2 (Plan Design)

Producer's Risk Quality (PRQ) (%)

0.5 — 3.5 — 10

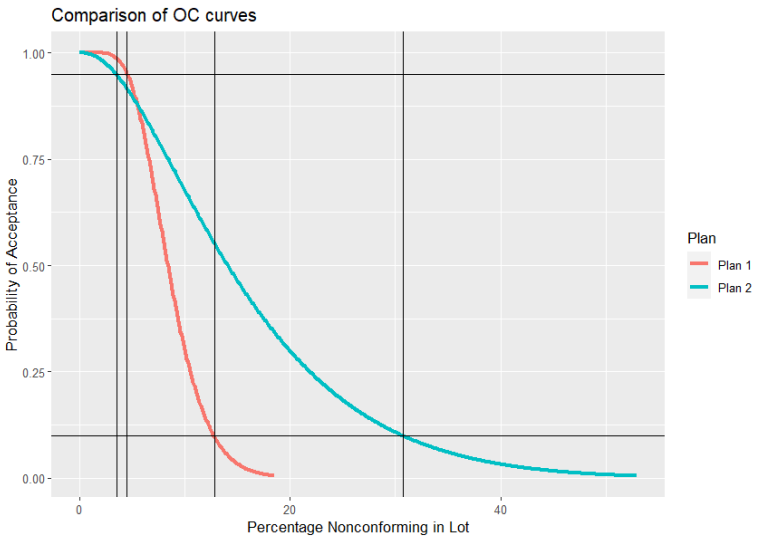
0.5 1.5 2.5 3.5 4.5 5.5 6.5 7.5 8.5 9.510

Consumer's Risk Quality (CRQ) (%)

5 — 35 — 40

5 9 13 17 21 25 29 33 37 40

Plan Details and OC | Risk Settings



Plan	n	k	PRQ	PR	CRQ	CR
Plan 1	28.00	1.38	4.50	0.05	12.80	0.10
Plan 2	5.00	1.08	3.50	0.05	35.00	0.10

1.2 Selection of options for sampling plans

A. Determine sampling plan options

Step 1. Type of data

Are the test results expressed as pass/fail outcomes (or equivalent) or are they measurements?

Pass/Fail (or equivalent) outcomes (Attributes)	Go to step 2
Measurements (variables)	Go to step 3

Help on attributes data

Help on variables data

Step 2. Attributes data

Is the inspection error negligible or non-negligible?

Negligible	\\	PR & CR	
	CXG 50 4.2.3	CR only	ISO2859-2
	CXG 50 Appendix II	PR only	ISO2859-1
Non-negligible			
	CXG 50 5.1.1	Known Inspection errors	

Help on Design of Attributes Plans

Step 3. Variables data

Does the provision relate to compliance of the distribution or to the average level of the characteristic?

Step 3.a. Plans to assess compliance of the distribution

Is the characteristic normally distributed, a compositional characteristic or does it follow some other distribution?

Normally distributed	Go to step 4
Compositional proportion	Go to step 6
Some other distribution	Go to step 7

Help on Design of Variables Plans

Step 3.b. Plans for the average level

Plans for the average level	Go to step 8

Help on provision

Help on average level

Step 4. Variables plans, normally distributed characteristics

Is measurement uncertainty negligible or non-negligible?

Negligible	CXG 50 4.3.3	PR & CR	
	CXG 50 4.3.4	CR only	ISO3951-6
	CXG 50 Appendix 2	PR only	ISO3951-1
Non-negligible	Go to step 5		

Step 5. Variables plans, normally distributed characteristics, non-negligible measurement uncertainty

Is the measurement uncertainty normally distributed or does it follow some other distribution?

Normally distributed	CXG 50 5.2.7	PR & CR	
	CXG 50 5.2.5	CR only	ISO3951-6
Some other distribution	CXG 50 5.2.8	PR & CR	

Step 6. Compositional proportions

Is measurement uncertainty negligible or non-negligible?

Negligible	CXG 50 4.4.10	PR & CR

Non-negligible	Go to step 5

Step 7. Characteristic is neither normally distributed nor a compositional proportion

Is the measurement uncertainty negligible or non-negligible?

Negligible	CXG 50 4.2.7	PR & CR
Non-negligible	CXG 50 5.2.8	PR & CR

Step 8. Provision is expressed in terms of the average level in a lot

Is the measurement uncertainty negligible or non-negligible?

Negligible	CXG 50 4.4.8	PR & CR

Non-negligible

[no information provided]

B. Specify stringency for the sampling plan (plans to assess compliance to minimum or maximum levels)

Consumer's risk quality level (CRQ)

What percentage nonconforming (quality level?) would you allow in lots that you would want to reject most of the time?	6.5%

Consumer's risk (CR)

What consumer's risk are you prepared to allow, i.e. how often would you want to accept lots containing 6.5 percent nonconforming?	10%

If the characteristic is a 'serious' food safety (or other) concern:

• it might not be appropriate to control producer's risks explicitly;

• use ISO plans (or alternatives) that control only the consumer's risk.

If the characteristic is not a 'serious' food safety or other concern, it is appropriate to also control the producer's risk.

Producer's risk quality level (PRQ)

What percentage nonconforming (quality level?) would need to be present in lots that you would want to accept most of the time?	5%

Producer's risk (PR)

What producer's risk are you prepared to allow, how often would you want to reject lots containing 5 percent nonconforming?	5%

C. Evaluate plan to determine plan parameters and calculate operating characteristic

Determine the number of samples and the acceptance number (attributes plans) or the acceptability constant (variables plans)

Supporting material

Context	Term	Explanation
Nature of the provision	Provision	A provision is a requirement for a commodity that must be met in order that the commodity conforms to the standard.
Nature of the provision	Overall distribution	Specification limits may be expressed as a minimum or a maximum limit (or both) applied to either the overall distribution of the characteristic in the lot, e.g. the percentage nonconforming quality level, or to the average level.
Nature of the provision	Average level	In some cases, such as the net weight of packages, a limit is set on the average level, with the intention that the average level in the batch should not be less than the limit. In Codex, although an example of sampling plans for bulk materials, the plans for aflatoxins are also based on compliance of the average level, to ensure that there is a small chance that the average level in a lot exceeds the maximum limit. It is usually assumed that the quality characteristic is normally distributed; the appropriateness of the distribution is less critical when compliance of the average level is being assessed. It is also usually assumed that there is a single specification limit, either a lower specification limit, L or an upper specification limit, U.
Types of data	Attributes	Data for which the test results have nominal outcomes or are measured on a scale, particularly binary outcomes such as pass or fail, and measurements classified as binary outcomes.
Types of data	Variables	Inspection by variables means that the outcomes of the measurements on each sample is a number, usually a decimal number. This is in contrast to attributes data where pass/fail outcomes are obtained or on a scale (sometimes described numerically, e.g. 1–5).

Type of sampling plan	Attributes plan	Inspection by attributes consists of examining an item, or characteristics of an item, and classifying the item as 'conforming' or 'nonconforming'. The action to be taken is decided by counting the number of nonconforming items or the number of nonconformities found in a random sample. An inspection by attributes sampling plan specifies the number of samples (n) and the maximum number of nonconforming items, referred to as the acceptance constant (c), for the lot to be accepted. The values of n and c are worked out from the specified levels of allowable risk.
Type of sampling plan	Variables plan	Inspection by variables plans use means and standard deviations (SD) calculated from the measurements (variables data) to make a decision about the acceptance of a lot. These plans are specified by the number of samples required to be taken (n) and an acceptability constant (k).
Measurement uncertainty		Parameter, associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand (i.e. the quantity intended to be measured). Measurement can consist of random and systematic components.
Lot standard deviation		A parameter, usually expressed as a SD, describing the variation of a characteristic within a lot.
Negligible measurement uncertainty		The situation where the measurement uncertainty (MU) is small in relation to the lot SD and does not need to be taken into account in the design of a sampling plan. Typically, MU is considered negligible if the SD representing the MU is less than 10 percent of the lot SD.
Non-negligible measurement uncertainty		Refers to cases where the MU is NOT negligible.
Standard deviation		SD is a measure of the amount of variation or dispersion in a set of values.
Known (true) standard deviation		Conceptually, the SD that would be found, for example, if every item in a lot was measured. In practice, standard deviations can be considered known if calculated using a reasonably large number of test results, typically 100–200. For a SD representing the longer-term variation of a process to be considered known, the process must be stable (consistent) over time.
Estimated (sample) standard deviation		A SD calculated from a smaller amount of data than required for the SD to be considered known.
Normal distribution		A statistical distribution commonly used in many branches of statistics to describe the variation of a measurement method under certain conditions or of a characteristic within a lot. A normal distribution is described by its mean (i.e. average level) and SD and follows a characteristic 'bell-shaped' curve.
Compositional proportion		A characteristic whose concentration within a lot can be expressed as a 'mass fraction', a number taking values between zero and one. Strictly speaking compositional proportions are dimensionless, and do not have proper units

		of measure, although it is common to express then using units such as percentages, parts-per-million (ppm) etc.
Producer's risk	PR	In general terms, PR is the risk that a lot of good quality will be rejected. More specifically, in the design of acceptance sampling plans, producer's risk is the probability of rejecting a lot that has a quality level equal to the producer's risk quality (PRQ) level.
Producer's risk quality level	PRQ	The quality level (percentage nonconforming in the lot) at which the probability of rejecting the lot is equal to the specified producer's risk (PR).
Consumer's risk	CR	Consumer's risk (CR) is the risk that a lot of poor quality will be accepted. More specifically, in the design of acceptance sampling plans, consumer's risk is the probability of accepting a lot that has a quality level equal to the consumer's risk quality (CRQ) level.
Consumer's risk quality level	CRQ	The quality level (percentage nonconforming in the lot) at which the probability of accepting the lot is equal to the specified CR.

APPENDIX II

ISO INSPECTION PLANS INDEXED BY PRODUCER'S RISK

1 ISO Inspection plans indexed by producer's risk – Introduction/Background

As noted in sections 4.2.3 and 4.3.4, the sampling plans included in the ISO 2859[Error! Bookmark not defined.] and ISO 3951[Error! Bookmark not defined.] standards differ from plans discussed elsewhere in these guidelines in that they have been designed to explicitly control either the producer's risk (PR) or the consumer's risk (CR), but not both, and use a lot size relationship to determine the required sample size.

1.1 Lot size versus sample size

Statistically, the lot size does not have an important role in determining protection to consumers and producers, whereas changes in the sample size does affect the protection afforded by any plan.

However, despite this, a lot size versus sample size relationship has been built into the design of the sampling plans appearing in the ISO standards. This relationship is arbitrary, although it has the general effect of reducing the risks of making incorrect decisions for larger lots, where the costs incurred from incorrect decisions will be greater. This relationship means that the ISO standards are applicable only to lots that consist of discrete items.

As a consequence of employing the sample size versus lot size relationship, ISO has designated that sampling plans indexed by producer's risk quality (PRQ), explicitly controlling the producer's risk, are intended for the inspection of a continuing series of lots and plans indexed by consumer's risk quality (CRQ), explicitly controlling consumer's risk, as being suitable for the inspection of isolated lots. However, this distinction is no longer relevant if both types of risk are considered in the design of plans.

1.2 Sampling schemes

The ISO standards indexed by PRQ employ sampling schemes, sets of sampling plans with different levels of inspection to ensure quality is effectively controlled. Sampling schemes employ switching rules for changing between inspection levels based on recent quality history. Typically, and in ISO standards, switching occurs between normal, tightened, and reduced inspection plans within each sampling scheme:

- normal inspection is used when the process is considered to be operating at, or slightly better than, the PRQ;
- tightened inspection uses stricter decision rules than those used in normal inspection. The main objective of using tightened inspection is to exert pressure on the producer when the quality is poorer than the PRQ by introducing a higher rate of rejection; and
- reduced inspection permits smaller sample sizes than those used in normal inspection. When the level of the submitted quality is sufficiently good, reduced inspection offers sampling economy.

Sampling schemes provide more comprehensive assurance than the use of individual sampling plans. However, switching rules are considered too complex to apply in international trade, and from a consumer's point of view in general, although it is possible to design a sampling plan that controls the producer's and consumer's risks to the same levels as an overall sampling scheme.

1.3 *Table: Inspection by attributes plans in accordance with ISO 2859-1*[Error! Bookmark not defined.]

		Inspection level					
Lot size	**AQL**	**reduced**		**normal**		**tightened**	
(number of packages, each containing 1 or more units)		***n***	***c***	***n***	***c***	***n***	***c***
2 – 8	0.65%	8	0	8	0	8	0
	2.50%	2	0	5	0	8	0
	6.50%	2	0	2	0	3	0
9 - 15	0.65%	8	0	15	0	15	0
	2.50%	2	0	5	0	8	0
	6.50%	2	0	2	0	3	0
16 - 25	0.65%	8	0	20	0	25	0
	2.50%	2	0	5	0	8	0
	6.50%	5	1	8	1	13	1

		Inspection level					
Lot size	**AQL**	**reduced**		**normal**		**tightened**	
26 – 50	0.65%	8	0	20	0	32	0
	2.50%	2	0	5	0	8	0
	6.50%	5	1	8	1	13	1
51 - 90	0.65%	8	0	20	0	32	0
	2.50%	13	1	20	1	32	1
	6.50%	5	1	13	2	13	1
91 - 150	0.65%	8	0	20	0	32	0
	2.50%	13	1	20	1	32	1
	6.50%	8	2	20	3	20	2
151 – 280	0.65%	8	0	20	0	32	0
	2.50%	13	1	32	2	32	1
	6.50%	13	3	32	5	32	3
281 – 500	0.65%	50	1	80	1	125	1
	2.50%	20	2	50	3	50	2
	6.50%	20	5	50	7	50	5
501 – 1 200	0.65%	50	1	80	1	125	1
	2.50%	32	3	80	5	80	3
	6.50%	32	6	80	10	80	8
1 201 – 3 200	0.65%	50	1	125	2	125	1
	2.50%	50	5	125	7	125	5
	6.50%	50	8	125	14	125	12
3 201 - 10 000	0.65%	80	2	200	3	200	2
	2.50%	80	6	200	10	200	8
	6.50%	80	10	200	21	200	18
10 001 – 35 000	0.65%	125	3	315	5	315	3
	2.50%	125	8	315	14	315	12
	6.50%	80	10	200	21	200	18
35 001 – 150 000	0.65%	200	5	500	7	500	5
	2.50%	200	10	500	21	500	18
	6.50%	80	10	200	21	200	18
150 001 – 500 000	0.65%	315	6	800	10	800	8
	2.50%	200	10	500	21	500	18
	6.50%	80	10	200	21	200	18
500 001 and over	0.65%	500	8	1250	14	1250	12
	2.50%	200	10	500	21	500	18
	6.50%	80	10	200	21	200	18

If sample size n equals, or exceeds, lot size, carry out 100 % inspection.

1.4 Table: Inspection by variables plans from ISO 3951-1 (lot SD unknown)

		Inspection level					
Lot size	**AQL**	**reduced**		**normal**		**tightened**	
(number of packages, each containing 1 or more units)		***n***	***k***	***n***	***k***	***n***	***k***
2 – 8	0.65%	6	1.476	8	1.889	8	2.079
	2.50%	4	0.850	4	1.242	6	1.476
	6.50%	4	0.586	4	0.735	3	0.950
9 - 15	0.65%	6	1.476	11	1.889	15	2.079
	2.50%	4	0.850	4	1.242	6	1.476
	6.50%	4	0.586	4	0.735	3	0.950
16 - 25	0.65%	6	1.476	11	1.889	15	2.079
	2.50%	4	0.850	4	1.242	6	1.476
	6.50%	4	0.586	6	0.939	6	1.061
26 – 50	0.65%	6	1.476	11	1.889	15	2.079
	2.50%	4	0.850	9	1.323	6	1.476
	6.50%	4	0.586	6	0.887	9	1.218
51 - 90	0.65%	6	1.476	11	1.889	15	2.079
	2.50%	6	1.061	13	1.475	13	1.569
	6.50%	5	0.550	9	0.869	9	1.190
91 - 150	0.65%	6	1.476	11	1.889	15	2.079
	2.50%	9	1.218	13	1.426	18	1.682
	6.50%	7	0.507	14	0.935	14	1.147
151 – 280	0.65%	11	1.642	22	1.972	15	2.079
	2.50%	9	1.190	20	1.411	18	1.659
	6.50%	9	0.628	21	0.945	21	1.227
281 – 500	0.65%	17	1.769	30	2.079	28	2.153
	2.50%	14	1.147	30	1.471	27	1.636
	6.50%	14	0.601	33	1.036	32	1.225
501 – 1 200	0.65%	23	1.893	31	2.061	38	2.263
	2.50%	21	1.227	46	1.482	41	1.702
	6.50%	21	0.830	52	1.120	50	1.245
1 201 – 3 200	0.65%	24	1.862	48	2.043	40	2.237
	2.50%	32	1.225	69	1.552	63	1.702
	6.50%	33	0.954	79	1.195	78	1.281
3 201 - 10 000	0.65%	37	1.853	71	2.101	61	2.230
	2.50%	48	1.394	105	1.619	99	1.720
	6.50%	52	1.120	124	1.239	122	1.325
10 001 – 35 000	0.65%	54	1.904	108	2.104	89	2.279
	2.50%	71	1.489	159	1.683	150	1.752
	6.50%	52	1.120	124	1.239	122	1.325
35 001 – 150 000	0.65%	84	1.914	159	2.166	137	2.285
	2.50%	105	1.619	247	1.716	233	1.785
	6.50%	52	1.120	124	1.239	122	1.325
150 001 – 500 000	0.65%	117	2.037	239	2.220	214	2.300
	2.50%	105	1.619	247	1.716	233	1.785

		Inspection level					
Lot size	**AQL**	**reduced**		**normal**		**tightened**	
	6.50%	52	1.120	124	1.239	122	1.325
500 001 and over	0.65%	169	2.117	348	2.268	323	2.324
	2.50%	105	1.619	247	1.716	233	1.785
	6.50%	52	1.120	124	1.239	122	1.325

If sample size n equals, or exceeds, lot size, carry out 100 % inspection.

NOTES

[1] FAO and WHO. 2009. *Guidelines on Analytical Terminology*. Codex Alimentarius Guideline, No. CXG 72-2009. Codex Alimentarius Commission. Rome.
[2] ISO.1999. *ISO 2859: Sampling procedures for inspection by attributes – Part 1: Sampling schemes indexed by acceptance quality limit (AQL) for lot-by-lot inspection*. Geneva. ISO.
[3] ISO. 2022. *ISO 3951-1: Sampling procedures for inspection by variables – Part 1: Specification for single sampling plans indexed by acceptance quality limit (AQL) for lot-by-lot inspection for a single quality characteristic and a single AQL*. Geneva. ISO.
[4] FAO and WHO. 2004. *Guidelines on Measurement Uncertainty*. Codex Alimentarius Guideline, No. CXG 54-2004. Codex Alimentarius Commission. Rome.
[5] FAO and WHO. 2013. *Principles for the Use of Sampling and Testing in International Food Trade*. Codex Alimentarius Guideline, No. CXG 83-2013. Codex Alimentarius Commission. Rome.
[6] EURACHEM & CITAC. 2000. *Guide quantifying uncertainty in analytical measurement (Second Edition)*. EURACHEM Secretariat. BAM. Berlin. www.eurachem.org
[7] ISO. 2020.*ISO 2859-2: Sampling procedures for inspection by attributes – Part 2: Sampling plans indexed by limiting quality (LQ) for isolated lot inspection*. Geneva. ISO.
[8] ISO. forthcoming. *ISO/DIS 3951-6: Sampling procedures for inspection by variables – Part 6: Specification for single sampling plans for isolated lot inspection indexed by limiting quality (LQ)*. Geneva. ISO.
[9] FAO and WHO. 1995. *General standard for Contaminants and Toxins in food and feed*. Codex Alimentarius Standard, No. CXS 193-1995. Codex Alimentarius Commission. Rome.
[10] ISO. 2017. *ISO/IEC 17025: General requirements for the competence of testing and calibration laboratories*. Geneva. ISO.
[11] ISO. 2019. *ISO 5725-2: Accuracy (trueness and precision) of measurement methods and results – Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*. Geneva. ISO.
[12] ISO. 1994. *ISO 5725-1: Accuracy (trueness and precision) of measurement methods and results – Part 1: General principles and definitions*. Geneva. ISO.
[13] ISO. 1994. *ISO 5725-3: Accuracy (trueness and precision) of measurement methods and results – Part 3: Intermediate measures of the precision of a standard measurement method*. Geneva. ISO.

APPENDIX V

Guidance to select Type II methods from multiple Type III methods

(For inclusion in the Information Document: Comprehensive guidance for the process of submission, consideration and endorsement of methods for inclusion in CXS 234 – section 3.7bis)

Introduction

It is not uncommon that several analytical methods are proposed for a single commodity – provision combination. However, only one of these can be designated as the reference method (Type II method). The following paragraphs give guidance on the selection of a Type II method from multiple Type III methods.

Codex Methods of Analysis

According to the Procedural Manual, the Codex analytical methods are primarily intended as international methods for the verification of provisions in Codex standards. They should be used for reference, in calibration of methods in use or introduced for routine testing and control purposes.

Purpose of Reference Methods (Type II)

Definition as per the Procedural Manual: A Type II method is the one designated Reference Method where Type I methods do not apply. It should be selected from Type III methods (as defined below). It should be recommended for use in cases of dispute and for calibration purposes.

Purpose of Alternative Approved Methods (Type III)

As per description in the Procedural Manual, a Type III method is one which meets the criteria required by the Committee on Methods of Analysis and Sampling for methods and may be used for control, inspection or regulatory purposes.

In the event of multiple Type III methods for the same provision-commodity combination, it is expected that these methods, although they might use different approaches, should result in equivalent decisions (compliant vs. non-compliant).

Considerations for choosing a Type II method among multiple Type III methods.[1]

- As the scope of methods of analysis are aligned with various matrices from many groups of commodities (Codex Procedural Manual, General Criteria for the Selection of Methods of Analysis, Recommended Methods of Analysis and Sampling (CXS 234-1999)), the method explicitly validated for the commodity stated in the Codex provision should be preferred: e.g. if a method for copper in infant formula is required, a method specifically validated for this commodity should be preferred to a method validated for milk powder.
- The method validated for more than one matrix from a specific commodity[2] should be preferred. E. g. a method validated for milk-based, and soy protein-based infant formulae should be preferred to a method validated only for milk-based infant formula.
- The method with the best selectivity should be preferred.
- The method with the best precision data (if this precision difference is relevant to the question asked) should be preferred.
- The method where a certified reference material, preferably from a matrix similar to that used in the scope of the method, was included in the validation should be preferred.
- The method should be practicable and applicable under normal laboratory conditions.

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1 In some situations, CCMAS may decide not to apply these selection considerations, e.g. for ethical, economic or safety reasons. This decision must be duly justified.

2 Different matrices belonging to one commodity. E.g. infant formula includes milk-based, soy-based, hydrolyzed protein based.

Appendix I

Validation of the proposed decision guidance

To test the proposed selection guidance, the following commodity-provision combinations with multiple Type III methods included in CXS-234 were used:

- Sodium and Potassium in infant formula (1 Type II and 3 Type III methods)
- Copper in milkfat products (1 Type II, 2 Type III methods)

Table 1: Selection guidance for Type II methods

Provision and Commodity	Method	Principle	Type	Type II				
				validated for commodity	validated for larger panel matrices	best selectivity	best precision data	certified reference material included pref. Similar matrix scope
Sodium/ Potassium in infant formula	AOAC 2015.06	ICPMS	Type II	x	x		x	x
	AOAC 2011.14	ICPOES	Type III	x	x			x
	ISO 8070 IDF 119	FAAS	Type III Was Type II	no, milk products only	x			x milk powder
	AOAC 986.24	ICPOES	Was Type III					?
Copper in milkfat products	AOAC 2015.06	ICPMS	Type II	yes, butter	x	x		yes, infant formula
	ISO 5738 IDF 76	photometry	Type III	yes, butter, butterfat	x		x	no
	AOAC 2011.14	ICPOES	candidate Type III	yes, butter	x			yes, infant formula

Considerations selection Type II method Sodium/Potassium in infant formula:

- AOAC 986.24 cannot be considered as Type II because of difference in analytical steps as compared to other Type III methods, which may have implications on the results. In addition, this method has 'Safety concerns' (Perchloric acid destruction). Method is rightfully revoked by the SDO and CXS-234.
- ISO 8070 | IDF 119, has an option to use dry ashing as a sample preparation, which is not appropriate for the determination of sodium. In addition, the method is not validated for Infant Formula. In conclusion, this method has several drawbacks as compared to the other 2 candidate Type II methods: AOAC 2011.14 and AOAC 2015.06.
- Comparing AOAC 2015.06 and AOAC 2011.14, which are both validated on the same samples, AOAC 2015.06 has better precision data and therefore should be preferred as Type II method. (MAS40/ CRD05 for precision data)

Considerations selection Type II method Copper in milkfat products:

- The validation of AOAC 2011.14 does not cover the range of the provision and consequently cannot be considered as Type III method. (MAS40/CRD06 for precision data)
- Although ISO 5738 | IDF 76 based on photometry seems to have better precision data, AOAC 2015.06 based on ICP-MS has a better selectivity and therefore should be preferred as Type II.

Conclusions

From the examples of Sodium and Potassium in infant formula and Copper in milkfat products, the proposed guidance is suitable for the selection of the appropriate Type II method when multiple Type III methods exist, and may therefore support CCMAS in the process of consideration and endorsement of methods for inclusion in CXS234.