CODEX ALIMENTARIUS COMMISSION

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Food and Agriculture Organization of the United Nations

World Health Organization

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REP25/MAS

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX ALIMENTARIUS COMMISSION

Forty-eighth Session
10-14 November 2025

REPORT OF THE 44th SESSION OF THE CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

Virtual

5 – 8 May and 14 May 2025

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LIST OF ABBREVIATIONS

(T)CD	(Thermal) Conductivity Detector
102NP method	Method in Appendix XI of CXS 234-1999
AACC	AACC International
AOAC	AOAC International (formerly known as Association of Official Agricultural Chemists)
App(s)	Application(s)
CAC	Codex Alimentarius Commission
CCAFRICA	FAO/WHO Coordinating Committee for Africa
CCCF	Codex Committee on Contaminants in Foods
CCEXEC	Executive Committee of the Codex Alimentarius Commission
CCFA	Codex Committee on Food Additives
CCFFP	Codex Committee on Fish and Fishery Products
CCFL	Codex Committee on Food Labelling
CCMAS	Codex Committee on Methods of Analysis and Sampling
CCNASWP	FAO/WHO Coordinating Committee for North America and the South West Pacific
CCNFSDU	Codex Committee on Nutrition and Foods for Special Dietary Uses
CCPFV	Codex Committee on Processed Fruits and Vegetables
CEN	European Committee for Standardization
CL(s)	Circular letter(s)
CRD	Conference room document
CXG	Codex guideline
CXS	Codex standard
ELISA	Enzyme Linked Immunosorbent Assay
EN	European standard
ENV	European prestandard
EU	European Union
EWG	Electronic working group
FAO	Food and Agriculture Organization of the United Nations
FDA	Food and Drug Administration
GC	Gas chromatography
GSFA	*General standard for food additives*
IAM	Inter-Agency Meeting
ICA	International Confectionery Association
ICC	International Association for Cereal Science and Technology
ICUMSA	International Commission for Uniform Methods of Sugar Analysis
IDF	International Dairy Federation

IFU	International Fruit and Vegetable Juice Association
ISO	International Organization for Standardization
LOD	Limit of determination
LOQ	Limit of quantification
ML(s)	Maximum level(s)
MoU	Memorandum of understanding
MS	Mass spectrometry
NFCSO	National Food Chain Safety Office
NPC	Numeric performance criteria
Nx	Nitrogen to protein conversion factor(s)
PAL	Precautionary allergen labelling
PWG	Physical working group
QTOF	Quadrupole Time-of-Flight
SDO(s)	Standard development organization(s)
TC	Technical committee
TFFJ	*ad hoc* Intergovernmental Task Force on Fruit and Vegetable Juices
UK	United Kingdom
USA	United States of America
USPC	United States Pharmacopeial Convention
VWG	Virtual working group
WHO	World Health Organization

LIST OF CONFERENCE ROOM DOCUMENTS (CRDs)

CRD No.	Agenda Item	Submitted by
1	Division of Competence	European Union
2 Rev.1	3	Chair of the Virtual Working Group (Australia)
3	10	Chair of the Inter-Agency Meeting (IAM)
4	3	AACC International (AACC), AOAC International (AOAC) and International Association for Cereal Science and Technology (ICC)
5	3	AOAC International (AOAC), International Dairy Federation (IDF) and International Organization for Standardization (ISO)
6	3	Norway
7	3	Codex Secretariat
8	2	Codex Secretariat
9	4	International Association for Cereal Science and Technology (ICC)
10	3	Brazil
11	4.1, 4.2, 6.1	El Salvador
12	4.2	International Dairy Federation (IDF) and International Organization for Standardization (ISO)
13	5.1, 5.2	Philippines
14	3.1, 5.1	Senegal
15	4.2	European Union and Japan
16 Rev.1	3.2, 6.2, 9	Thailand
17 Rev.1	2.1	New Zealand
18	9	Inter-Agency Meeting (IAM)
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20	4.2	Australia
21	11	Algeria
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30	1, 2, 2.1, 3.1, 3.2, 4.1, 4.2, 5.1, 5.2, 6.1, 6.2, 7, 8, 9	United Republic of Tanzania

CRD No.	Agenda Item	Submitted by
31	3.1, 6.1	Azerbaijan
32	1, 2, 2.1, 3.1, 3.2, 4.1, 4.2, 5.1, 5.2, 6.1, 6.2, 7, 8, 9	Kenya
33	5.1	International Fruit and Vegetable Juice Association (IFU)
34	5.1, 5.2, 6.1, 8	International Union of Food Science and Technology (IUFoST)
35	6.1	Germany and New Zealand
36	3.1, 3.2, 5.2, 6.1, 6.2, 7	Chile
37	5.1	Germany and International Fruit and Vegetable Juice Association (IFU)

INTRODUCTION

1. The Codex Committee on Methods of Analysis and Sampling (CCMAS) held its 44th Session virtually from 5 to 8 May and on 14 May 2025, 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, Head of Department, Department of Digital Food Science, University of Veterinary Medicine, Budapest acted as the Vice-Chairperson. The Session was attended by 74 Member countries, one Member organization and 21 Observer organizations. The list of participants is contained in Appendix I.

OPENING OF THE SESSION

2. Dr Beáta Felkai, Deputy State Secretary of the Ministry of Agriculture of Hungary, opened the session (via video message) and extended her warmest welcome to all participants. Dr Felkai highlighted that food regulations and an international regulatory framework are essential in response to the globalization of food trade, technological advances, and consumer awareness and emphasised that it was critical for Members to exchange national laboratory findings and develop laboratory related activities together. Dr Felkai further expressed that the high level of participation in CCMAS was testament to the importance of its work.

3. Dr Haris Hajrulahovic, the World Health Organization (WHO) Representative and Head of Country Office, Ms Mary Kenny, Food Safety and Consumer Protection Officer, the Food and Agriculture Organization of the United Nations (FAO) Regional Office for Europe and Central Asia, and Dr Allan Azegele, Chairperson of the Codex Alimentarius Commission (CAC) (via video message) also addressed the Committee.

Division of Competence

4. CCMAS44 noted the division of competence between the European Union and its Member States, according to paragraph 5, Rule II of the Procedure of the CAC.

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

5. CCMAS44 adopted the Provisional Agenda as the Agenda for the Session and noted that the methods of analysis highlighted in conference room document (CRD21) were already contained in CRD07 and would be considered under Agenda item 3 (Endorsement of methods of analysis provisions and sampling plans in Codex standards).

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

6. The Codex Secretariat introduced the item and recalled that some matters from the CAC47, the 86th and 87th Sessions of the Executive Committee of the Codex Alimentarius Commission (CCEXEC86 and CCEXEC87), the 36th Session of the Codex Committee on Fish and Fishery Products (CCFFP36), the 44th Session of the Codex Committee on Nutrition and Foods for Special Dietary Uses (CCNFSDU44) and the 25th Session of the FAO/WHO Coordinating Committee for Africa (CCAFRICA25) were for information purposes; and the following were considered by the virtual working group (VWG) meeting that met on 29 and 30 April and the in-session VWG that met on 5 May 2025:

- Reply from CCFFP36 on the intended use of the amino acid nitrogen provision in the *Standard for fish sauce* (CXS 302-2011);
- Reply from CCAFRICA25 on the determination of chloride in dried meat in *the Regional standard for dried meat* (Africa) (CXS 350R-2022);
- Request from the 48th Session of the Codex Committee on Food Labelling (CCFL48) on guidance relating to methods of analysis for precautionary allergen labelling (PAL); and
- Request from 55th Session of the Codex Committee on Food Additives (CCFA55) on the testing method for sodium chloride in food grade salt in the *Standard for food grade salt* (CXS 150-1985).

7. CCMAS44 further noted that the 17th Session of the FAO/WHO Coordinating Committee for North America and the South West Pacific (CCNASWP17) was revising the draft standard operating procedure on the qualitative assessment of kava varieties for nobility in the *Regional standard for kava products for use as a beverage when mixed with water* (North America and South West Pacific) (CXS 336R-2020) in response to CCMAS's request. This work was still in progress and no action from CCMAS was required at this time.

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1 CX/MAS 25/44/1

2 CX/MAS 25/44/2; CX/MAS 25/44/2-Add.1

Nitrogen to protein conversion factors for commodities approved by commodity committees

8. CCMAS44 recalled that CAC47 had not approved the list of nitrogen to protein conversion factors (Nx) for inclusion as an Annex in the *Recommended methods of analysis and sampling* (CXS 234-1999). CAC47 requested CCMAS to: i) update Part 3 of Appendix II of REP24/MAS and ensure consistency of Nx with those in commodity standards; ii) consider the request from CCNFSDU44 on the inclusion of the Nx for follow-up formula; and iii) resubmit the Annex to CAC for adoption in the future.

9. The Codex Secretariat explained that the list of Nx had been updated with assistance from Brazil and Chile and proposed that CCMAS44 consider the revised list presented in CRD08. The Codex Secretariat further explained that the list contained those Nx that had been agreed by relevant committees and were either referenced in commodity standards or in CXS 234-1999 and that the aim of the list was to provide clarity and accessibility, for ease of use by analysts.

10. In addition to some editorial corrections and additions to the list, CCMAS44 noted that there were inconsistencies with respect to the Nx for soy products. CCMAS44 noted that these factors would need review by the relevant commodity committees to ensure consistency. CCMAS44 also noted that the list would continue to be updated as new Nx were agreed by the respective commodity committees whose remit was to provide Nx. The Codex Secretariat explained that in cases where Nx had been developed by a committee adjourned *sine die* and where such Nx needed review, the Codex Secretariat would look into modalities for their review.

Conclusion

11. CCMAS44:

    i. noted the matters for information referred by CAC, CCEXEC, and other Codex subsidiary bodies (CCFFP, CCFL, CCNFSDU and CCAFRICA);

    ii. noted that the work was ongoing in CCNASWP in relation to the qualitative assessment of kava varieties for nobility to support the *Regional standard for kava products for use as a beverage when mixed with water* (North America and the South West Pacific) (CXS 336R-2020);

    iii. noted that the request from CCFL for advice on methods of analysis for PAL would be considered under Agenda item 8, and that the replies from CCFFP and CCAFRICA and the request from CCFA would be considered under Agenda item 3;

    iv. encouraged more Members to take leadership roles in committee working groups;

    v. encouraged Members and Observers to provide inputs to the monitoring framework for the Codex Strategic Plan 2026-2031; and

    vi. agreed to forward the list of Nx as amended (Appendix III) for approval by CAC48 for inclusion in CXS 234-1999 as an Annex, noting inconsistencies with the Nx for soy products and proposing that these factors should be reviewed in the future.

ENDORSEMENT OF METHODS OF ANALYSIS PROVISIONS AND SAMPLING PLANS IN CODEX STANDARDS (Agenda item 3)

12. CCMAS44 considered the recommendations on methods of analysis proposed for endorsement and other related matters as presented in CRD02 Rev.1.

13. CCMAS44 made the following decisions which are also presented in Appendix II of the report as follows.

Matters referred to the Committee by other subsidiary bodies (refer to Agenda item 2)

Codex Committee on Fish and Fishery Products (CCFFP36)

Method for determination of amino acid nitrogen in fish sauce

14. CCMAS44 noted the response from CCFFP36 and agreed to retain the method for determination of amino acid nitrogen in fish sauce, AOAC 920.04 and AOAC 920.03, in CXS 234-1999.

FAO/WHO Coordinating Committee for Africa (CCAFRICA25)

Method for the determination of chloride in dried meat

15. CCMAS44 endorsed AOAC 935.47 and AOAC 937.09B as Type III methods as recommended in CRD02 Rev.1, Appendix I, Table 2.

Codex Committee on Food Additives (CCFA55)

Method for determination of sodium chloride in food grade salt

16. CCMAS44 agreed to transfer the method for the determination of sodium chloride in food grade salt as described in the *Standard for food grade salt* (CXS 150-1985) with editorial amendments to CXS 234-1999 (Part A). In addition, CCMAS44 agreed to transfer the related sampling plan to CXS 234-1999 (Part B).

17. CCMAS44 noted that the sampling plan was currently incompatible with the provision for sodium chloride and would require further review and agreed that such review could be taken up later under the work on the review of sampling plans (see Agenda item 6.2). CCFA would be informed accordingly.

Other matters

18. CCMAS44 agreed with an update to the hyperlink in the footnote referencing the method for iodine in food grade salt in CXS 234-1999.

ENDORSEMENT OF METHODS OF ANALYSIS PROVISIONS IN CODEX STANDARDS (Agenda item 3.1)[3]

Codex Committee on Nutrition and Foods for Special Dietary Uses (CCNFSDU44)

Methods for provisions for Follow-up formula

19. CCMAS44 agreed to:

- endorse the methods as recommended in CRD02 Rev.1, Appendix II, Table 2;
- revoke and retype the methods as recommended in CRD02 Rev.1, Appendix II, Table 3;
- inform CCNFSDU that the methods of analysis submitted by them to CCMAS were forwarded for adoption by CAC48 and relevant methods of analysis would be revoked consequentially; and
- request CCNFSDU to consider:
    - whether it would be appropriate to develop numeric performance criteria (NPC) for methods of analysis for Type II and Type III methods; or
    - additional methods for follow-up formula to align with those for infant formula for common provisions (Appendix II, Part 3.2).

20. With regard to the recommendation for CCNFSDU to consider developing NPC, the chair of the VWG clarified that CCMAS's preference was for committees to consider this approach. CCMAS44 noted that this approach would provide flexibility for countries to choose methods that meet these criteria. This approach was in accordance with the Procedural Manual that preference should be given to set NPC.

21. CCMAS44 agreed that the name and format for principles for the corresponding methods for infant formula in CXS 234-1999 would not be aligned with those agreed for follow-up formula at this stage pending the ongoing discussion on harmonization of names and format for principles (see Agenda item 9).

22. The Codex Secretariat noted that the request to CCNFSDU was timely as CCNFSDU was in the process of reviewing all methods for provisions in standards under their purview to assess their fitness-for-purpose and to identify additional/replacement methods, or other corrections through an EWG.

Dietary fibre (CXS 234-1999, Table 6. "Methods of analysis for dietary fibre: Guidelines for use of nutrition and health claims (CXG 23-1997): Tables of conditions for claims")

23. CCMAS44:

- endorsed the methods as proposed in CRD02 Rev.1, Appendix II, Table 1 together with the footnote as proposed by CCNFSDU44;
- agreed to request CCNFSDU to clarify whether the footnote was applicable only to AOAC 2022.01/AACC 32-61.01/ICC Standard No. 191. If not, the footnote should apply to all methods in Table 6 of CXS 234-1999, or a subset of the methods in this table; and
- agreed to request CCNFSDU to consider the proposed amendment to footnote 2 in CXS 234-1999, Table 6 in accordance with paragraph 26 and, if CCNFSDU agrees, CCMAS will delete the footnote applied only to AOAC 2022.01/AACC 32-61.01/ICC Standard No. 191.

[3] CX/MAS 25/44/3

24. CCMAS44 considered the appropriateness of the footnote proposed by CCNFSDU44, "*Isolated, purified, and/or synthetic fibres captured by AOAC 2022.01/ICC Standard 191/AACC 32-61.01 that do not meet the Codex definition of dietary fibre in the Guidelines on nutrition labelling (CXG 2-1985) should be subtracted from the final measurement, where deemed appropriate by competent authorities"*, to the methods as proposed by CCNFSDU44. In doing so, CCMAS44 considered changes to the footnote and its application to other methods in CXS 234-1999, Table 6. However, it was noted that any changes would need further consultation with CCNFSDU.

25. CCMAS44 noted comments made that the footnote proposed by CCNFSDU44 was not necessary, as footnote 2[4] in CXS 234-1999, Table 6 already applied to all methods. However, if the footnote as proposed by CCNFSDU44 was necessary, it should apply to all methods in CXS 234-1999, Table 6 and be amended accordingly.

26. CCMAS44 agreed to propose to CCNFSDU an amendment to footnote 2 in CXS 234-1999, Table 6 which would address the concerns raised by several delegations that the footnote forwarded by CCNFSDU should be consistently applied to all methods in CXS 234-1999, Table 6. If CCNFSDU agreed to amend footnote 2, then the footnote forwarded by CCNFSDU to accompany AOAC 2022.01/AACC 32.61.01/ICC Standard No. 191 could be deleted. The recommended amendment was as follows:

> Two issues are left for national authorities: to include monomeric units 3-9 and which isolated or synthetic compounds have physiological benefit. (Refer to the *Guidelines on nutrition labelling* (CXG 2-1985)). **Isolated, purified, and/or synthetic fibres captured by the analysis that do not meet the Codex definition of dietary fibre in CXG 2-1985 should be subtracted from the final measurement, where deemed appropriate by competent authorities.**

ENDORSEMENT OF METHODS OF ANALYSIS: OTHER RELEVANT MATTERS ARISING FROM THE AMENDMENT OF CXS 234-1999[5]

27. CCMAS44:

- noted the matters for information in CX/MAS 25/44/3-Add.1 and CRD07;
- agreed to transfer the methods of analysis as recommended in CRD02 Rev.1, Appendix III, Table 2, from the commodity standards to CXS 234-1999, noting that a review of these methods would still be needed to ensure they were still fit-for-purpose;
- endorsed the methods of analysis and NPC as recommended in CRD02 Rev.1, Appendix III, Tables 3 and 5, including the consequential revocation of methods of analysis;
- requested the Codex Secretariat to amend relevant commodity standards by replacing the methods of analysis with the general reference to CXS 234-1999 as per the Procedural Manual;
- noted the editorial amendment to the principle for the example methods AOAC 2015.06 / ISO 21424 | IDF 243 that meet the numeric performance criteria for copper in milk fat products, and agreed to the removal of ISO 5738 | IDF 76 and AOAC 960.40 as example methods, as indicated in CRD02 Rev.1, Appendix III, Table 4;
- agreed to retain the methods of analysis in CRD02 Rev.1, Appendix III, Tables 1, 6, 7 and 8 in the respective commodity standards; and
- agreed to establish an EWG chaired by Canada working in English only, to review the methods in CRD02 Rev.1, Appendix III, Tables 1, 2, 6, 7, and 8. The EWG should prepare and submit its report to the Codex Secretariat at least 3 months prior to CCMAS45.

Conclusion

28. CCMAS44 agreed to:

i. submit the methods of analysis; NPC for methods to determine sodium chloride and salt in salted Atlantic herring and salted sprat, salted fish and dried salted fish of *Gadidae* family of fishes, and sturgeon caviar, for incorporation into CXS 234-1999; and additional editorial amendments or corrections for adoption / revocation by CAC48 (Appendix II, Part 1);

ii. inform the relevant committees of the respective decisions taken at the session (paragraphs 14-18);

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4 Two issues are left for national authorities: to include monomeric units 3 – 9 and which isolated or synthetic compounds have physiological benefit (Refer to the *Guidelines on nutrition labelling* (CXG 2-1985))

5 CX/MAS 25/44/3 Add.1

iii. request CCNSFDU to consider the recommendations in paragraphs 19 and 23; and

iv. re-establish the Physical Working Group (PWG) on methods endorsement chaired by the United States of America (USA) and co-chaired by Hungary, Japan and Uruguay, working in English, French and Spanish, to meet immediately prior to CCMAS45, to consider all methods of analysis and sampling submitted by Codex Committees for endorsement, the outcomes of the work of the EWG (see paragraph 27) including retyping of the ISO 1871 method for determining protein in quinoa (see Agenda item 4.1); the proposals on the workable packages: cocoa products and chocolates and sugars and honey (see Agenda item 5); and any other matters referred by other Codex Committees or submitted by Members and Observers.

MATTERS PENDING FROM CCMAS43 (Agenda item 4)

METHODS OF ANALYSIS FOR PROTEIN IN QUINOA (Agenda item 4.1)[6]

29. CCMAS44 recalled that CCMAS43 had endorsed the ISO 1871 method for determining protein in quinoa as Type IV, noting that the typing could be reconsidered if more information were provided, such as the specific chemicals used for the catalysts, different reagents and their concentrations, and method conditions. A circular letter, CL 2024/91-MAS, was issued requesting this additional information for consideration by the VWG.

30. The VWG considered the replies to CL 2024/91-MAS, but the information provided at that time did not allow the VWG to find consensus on retyping ISO 1871. During the VWG, ISO 20483 was also offered as an alternative Type I method for the determination of protein in quinoa, but it currently lacked validation data for pseudocereals such as quinoa. The Observer from ISO indicated at the VWG that quinoa could be added as a validated matrix in future and that ISO might consider taking up that work if needed.

Discussion

Re-typing of the ISO 1871 method for determining protein in quinoa

31. CCMAS44 noted that information on the ISO 1871 method had been submitted by seven Members in CRD19 to support the retyping of the method from Type IV to Type I. This information included the validation data of the ISO 1871 method, as well as the reagents, conditions, and catalysts used in the validation studies.

32. A Member proposed that the ISO 1871 method for determining protein in quinoa could be retyped to Type I with an explanatory footnote (e.g. regarding conditions, the catalysts and reagents used), noting that the same footnote would be consequentially applied to the ISO 1871 method for determining protein in tehena to ensure consistency. It was reiterated that CCMAS should apply the same decisions regarding the use of ISO 1871 for the determination of protein to tehena to ensure consistency.

33. Members supporting this proposal were of the view that CRD19 already provided sufficient basis to retype the ISO 1871 method for determining protein in quinoa from Type IV to Type I, because:

- CCMAS43 already agreed that the validation data for the ISO 1871 method was not in question[7];
- the validation studies were conducted by internationally recognized national metrology institutions with the biggest analytical capacities in their respective countries; and
- a single reference material for quinoa was used in the validation studies.

34. Members and Observers which did not support the proposal expressed these concerns:

- ISO 1871 currently included in CXS 234-1999 was a general guidance and not a step-by-step method, since the ISO standard did not specify conditions and chemicals. In accordance with the definition for a Type I method, a detailed analysis of the steps taken across the different validation studies would be necessary to retype ISO 1871 from Type IV to Type I, even though there were no issues with the validation data.
- CRD19 contained some variations in the methods, chemicals and volumes of reagents used during the validation studies, hence a further review involving ISO should be conducted to determine if the information was sufficient to support retyping ISO 1871 from Type IV to Type I. It was clarified that ISO did not have the mandate to revise the data in CRD19 during such a review.
- There was insufficient time for CCMAS44 to consider whether the conditions (e.g. reagents and catalysts) across different laboratory studies in CRD19 were sufficiently harmonized to support the formulation of an explanatory footnote.

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6 CL 2024/91-MAS; CX/MAS 25/44/4 (Comments of Argentina, Bolivia (Plurinational State of), Egypt, European Union, Indonesia, Peru and Saudi Arabia)

7 REP24/MAS paragraph 22(ii)

35. A view was expressed that although CRD19 was submitted fairly late, it should be reviewed in the spirit of transparency.

36. Noting the lack of consensus to retype the ISO 1871 method for determining protein in quinoa at this stage, CCMAS44 agreed that this topic should be deferred to the PWG on endorsement that would meet immediately prior to CCMAS45 to allow more time to review CRD19, and the ISO 1871 method for determining protein in quinoa should remain in CXS 234-1999 as Type IV at this time (Appendix II, Part 2).

Extension of the ISO 20483 method to quinoa

37. A Member, recalling that ISO 20483 was offered as an alternative method during the VWG, suggested that the necessary validation studies could be performed to determine if this method could be extended from cereals and pulses to a pseudocereal such as quinoa. This suggestion was supported by another Member.

38. Nevertheless, it was noted that the decision to extend ISO 20483 to quinoa fell under ISO's prerogative and not CCMAS. It was also possible that ISO might establish a separate method instead of extending ISO 20483.

Conclusion

39. CCMAS44 agreed to:

    i. retain the ISO 1871 method for determining protein in quinoa in CXS 234-1999 as a Type IV method, noting the reservation of Peru; and

    ii. request the PWG on endorsement to reconsider the retyping of the ISO 1871 method for determining protein in quinoa based on the information provided in CRD19.

DETERMINATION OF MOISTURE CONTENT IN WHEY POWDER (Agenda item 4.2)[8]

40. CCMAS44 used CRD02 Rev.1, Appendix IV, Table 1 as the basis for discussions. Delegations noted that the ISO 5537 | IDF 26 method already existed in CXS 234-1999 as a Type I method, and that the discussion was limited to whether the method in CXS 234-1999 Appendix XI for the determination of water (moisture) in whey powders (hereafter known as the 102NP method) could be endorsed as Type IV with a footnote.

41. CCMAS44 made the following change to the footnote in CRD02 Rev.1, Appendix IV, Table 1: The sentence 'If trading partners agree, the Type IV method can be used in international trade; otherwise ISO 5537 | IDF 26 should be used' was deleted, as any method can be used as long as it has been agreed upon by trading partners.

42. An alternative footnote was proposed for consideration:

> *In particular for powders with high natural lactose content such as whey powders, the CXS 234 Appendix XI – Moisture method at normal pressure (102±2ºC) has been exceptionally included as a co-existing Type IV method despite performance showing poorer precision and results which may not be consistent with those obtained with ISO 5537 | IDF 26 to take into account practical matters of limited accessibility to equipment and calibration of the Type I method. In a dispute situation the Type I method shall be used".*

43. However, this alternative footnote did not find favour, as the original footnote incorporating the amendment in paragraph 41 better reflected the compromise found during the VWG.

44. An Observer added that if the 102NP method were endorsed, the title and scope contained in CXS 234-1999, Appendix XI, would need to be consequentially amended to include whey powders.

Conclusion

45. CCMAS44 agreed to forward the 102NP method with the footnote as amended in paragraph 41 to CAC48 for adoption as a Type IV method (Appendix II, Part 1.5), for whey powder, on an exceptional basis, in line with Section 3.9 (v) of the information document "Comprehensive guidance for the process of submission, consideration and endorsement of methods for inclusion in CXS 234" and to make the consequential amendments to Appendix XI in CXS 234-1999 (see paragraph 44).

OTHER MATTERS

Determination of the particle size of milling products using sieve analysis for use on edible cassava flour and gari

46. The Codex Secretariat informed CCMAS44 that while the International Association for Cereal Science and Technology (ICC) Recommendation N. 207 was endorsed during CCMAS43, CCMAS43 had noted that the method of analysis might be subject to revision as appropriate sieve sizes were not included. Following

[8] CX/MAS 25/44/5

CCMAS43, ICC initiated a revision of Recommendation N. 207, so that it would be applicable to edible cassava flour and gari. This work was now in its final stage and ICC would inform the Codex Secretariat once the work is complete. No further amendment to CXS 234-1999 was necessary.

Conclusion

47. CCMAS44 thanked ICC for the update contained in CRD09 and acknowledged that no amendment to CXS 234-1999 was necessary.

REVIEW OF METHODS OF ANALYSIS IN CXS 234 (Agenda Item 5)

48. CCMAS44 recalled that the recommendations from the EWGs on the two workable packages: fruit juices and nectars, and cocoa products and chocolate had been considered by the VWG on endorsement. CCMAS44 considered the recommendations presented in CRD02 Rev.1.

FRUIT JUICES WORKABLE PACKAGE (Agenda item 5.1)[9]

49. Germany, as Chair of the EWG, introduced the item and summarized the background of the review of methods of analysis for fruit juices and nectars vis-à-vis the *General standard for fruit juices and nectars* (CXS 247-2006) and CXS 234-1999, the work process and key points of discussions in the EWG, and comments and questions submitted by Members and Observers in reply to CL 2025/16-MAS. The EWG Chair further noted that such comments and questions, including other comments submitted in CRDs, were considered by the VWG that met before this Session. Based on the discussions and recommendations of the VWG, as presented in CRD02, the EWG Chair, with the assistance of the International Fruit and Vegetable Juice Association (IFU), prepared CRD37 for consideration by the plenary session.

50. The EWG Chair indicated that comments and questions submitted in reply to CL 2025/16-MAS and those issues identified by the EWG remain for discussion by the plenary as follows:

51. Issues identified by the EWG:

- CEN/TC 174, the technical committee (TC) responsible for the European standard (EN) methods for fruit and vegetable juices and nectars listed in CXS 234-1999 (30 in total), was disbanded by the European Committee for Standardization (CEN) in 2023. No other TC in CEN has taken over the work on the EN standards for these commodities. Consequently, CEN disbanded/inactivated the methods. Therefore, the methods are no longer supported by CEN.
- The European Prestandard (ENV) methods ENV 12142 (1996) for determining the stable hydrogen isotope ratio of water and ENV 12141 (1996) for determining the stable oxygen isotope ratio of water (Sections 3.2 Quality Criteria and 3.3 Authenticity of CXS 247-2005) have been withdrawn as the TC(s) responsible for their development and maintenance have been disbanded by CEN. Therefore, the provisions to determine the quality and authenticity of fruit juices and nectars are no longer represented by an available method. IFU will shortly publish methods based on the CEN method. CCMAS should decide if the provision needs to be deleted, if it can be put on hold until the IFU methods are published, or if new methods should be endorsed via the endorsement process.
- IFU 42, the method for determining the carbon dioxide content (Section 4 Additives and Section 5 Processing aids in CXS 247-2005), is no longer available. CCMAS44 should decide whether to delete the provision or endorse a new method via the methods endorsement process.

52. Issues identified in reply to CL 2025/16-MAS:

- Where several “analytes” are covered by the same method, they should be listed and assessed as individual analytes.
- The absence of numerical provisions for quality and authenticity in CXS 247-2005 should be addressed by estimating the limit of quantification (LOQ) for each method and deriving an “estimated lowest or threshold level supported by method LOQ”.
- New methods should replace the unavailable methods and be endorsed via the endorsement process.
- The performance of some methods (e.g. LOQ, working range) should be verified as “fit-for-purpose”.
- The validated or limited scope of certain matrices/commodities should be verified as “fit-for-purpose”.

[9] CL 2025/16-MAS; CX/MAS 25/44/6; CX/MAS 25/44/6-Add.1 (Comments of Australia, Ecuador, Egypt, Ghana, Iran (Islamic Republic of), Iraq, Peru, Senegal, Thailand, Uzbekistan and International Commission for Uniform Methods of Sugar Analysis (ICUMSA))

- Some provisions (e.g. malic acid-D, citric acid, glucose-D, and fructose-D) could use commercial and proprietary methods (i.e. enzymatic methods).
- The term “saccharose” should be replaced with the more common term “sucrose”.
- IFU 42 for determining the carbon dioxide content should be removed.

53. The EWG Chair explained that:

- due to a lack of corresponding numeric provisions for quality and authenticity in CXS 247-2005 against which to evaluate the methods for fruit juices and nectars listed in CXS 234-1999, the VWG could not reach a consensus on their endorsement;
- as CXS 234-1999 also referred to methods of analysis for food additives used in fruit and vegetable juices and nectars, the VWG proposed that CCMAS44 consider analytical methods vis-à-vis maximum levels (MLs) established in the *General standard for food additives* (GSFA) (CXS 192-1995) for the food categories 14.1.2.1 “Fruit juice”, 14.1.2.2 “Vegetable juice”, 14.1.2.3 “Concentrates for fruit juice”, and 14.1.3.1 “Fruit nectar” of the GSFA. These methods are compiled in Annex 1 of CRD37 for consideration by the Committee; and
- the remaining methods for quality and authenticity in CXS 234-1999 without corresponding numerical provisions in CXS 247-2005 were recommended to be reviewed and assessed by an expert group on analytical methods on fruit/vegetable juices/nectars, who could assist CCMAS in determining whether the fruit juice methods were still “fit-for-purpose”. These methods are compiled in Annex 2 of CRD37.

54. Based on the above explanation, CCMAS44 agreed to consider those analytical methods for fruit juices and nectars with provisions (either included in CXS 247-2005 or CXS 192-1995) as presented in Annex 1 of CRD37.

Discussion

Annex 1 of CRD37

55. The Codex Secretariat recalled that the *ad hoc* Intergovernmental Task Force on Fruit and Vegetable Juices (TFFJ) was dissolved following completion of CXS 247-2005 and that CAC had allocated work on fruit juices and nectars to the Codex Committee on Processed Fruits and Vegetables (CCPFV), currently adjourned *sine die*. Therefore, CCMAS44 could review the analytical methods for fruit juices and nectars in CXS 234-1999 and make decisions about their retention (and update as appropriate), replacement, or revocation as deemed necessary.

56. CCMAS44 endorsed some methods, revoked others, and retained several EN methods for five years to allow transition, as these methods, although no longer supported by the relevant standard development organization (SDO), were still in use in some countries.

57. As regards the temporary retention of specific EN methods that were no longer available for use, IFU indicated that soluble solids were a critical determination for fruit juices and that method IFU 8 was identical to EN 12143 and thus supported the temporary retention of the EN method in CXS 234-1999. It was noted that a similar situation applied to EN methods for sucrose and phosphorus/phosphate. The Observer clarified that governments could access the IFU method for all EN methods for which there is an identical IFU method by signing a memorandum of understanding (MoU) with IFU. Upon signing an MoU, these methods can be provided without any need of purchasing.

58. CCMAS retained other methods for further consideration by the expert group due to a variety of reasons such as the unavailability of validation data at the time when the method was considered by the Committee (ascorbic acid, ISO 6557-2:1995); the validated limited scope of the method to support their application to all fruit juices (i.e. some matrices only) (citric acid, AOAC 986.13 (1996); citric/malic/quinic acid, AOAC 986.13 (1986)); whether the method was “fit-for-purpose” due to its sensitivity to check the required level, in particular presence/absence of the analyte (malic acid-D in apple juice, AOAC 995.06 (1998)).

Conclusion

59. CCMAS agreed to:

i. forward the analytical methods for fruit juices and nectars to CAC48 for endorsement or revocation as presented in Appendix II, Part 1.4; and

ii. retain other methods in CXS 234-1999 pending further consideration by the expert group (Appendix II, Part 3.3).

Annex 2 of CRD37

60. CCMAS44 further considered those analytical methods listed with no numerical provision in CXS 247-2005 that need a re-evaluation to determine whether they are still fit-for-purpose as presented in Annex 2 of CRD37.

61. CCMAS44 considered the opportunity to establish an expert group, under the leadership of IFU, that would review all methods listed in Annex 2 and those transferred from Annex 1. The expert group would report its findings to CCMAS45 for consideration and subsequent follow-up.

62. Regarding the proposal to establish this expert group, the Codex Secretariat clarified that IFU would be responsible for assembling it. The experts would be selected based on their capacity as experts in the field and not as representatives of their organizations or countries. The discussion, conclusions, and recommendations of the expert group would be submitted to CCMAS45 for consideration and follow-up as appropriate. A similar process was followed in the past for the initial groundwork by AACC International (AACC) on the cereals, pulses, and legumes workable package, where an EWG established by CCMAS followed up on the recommendations made by AACC. Codex Members interested in this work could approach IFU and nominate experts who would act in their capacity as experts.

63. IFU noted that due to the complexity of the issue, a structured approach was advisable, similar to the one taken by the TFFJ in the early 2000s during the preparation of the standard, and particularly the development of the list of analytical methods currently in CXS 234-1999. IFU would commit to assembling a diverse but focused group of global experts under its auspices. The group would be international and diverse but limited in size to maintain effectiveness. Experts would be selected based on expertise, not broad representation; only qualified experts would be included.

64. Some Members were concerned that reviewing methods without associated specifications or provisions would result in little or no progress. They highlighted that codes of practice exist in various regions, containing explicit provisions, specifications, and interpretations. They also requested clarification on whether the expert group would propose numerical values for provisions and match such provisions with the corresponding method to evaluate their fitness for purpose. Although they understood the magnitude of the task, considering the global nature of the standard for fruit juices and nectars, it was difficult to gauge how the expert group would carry out its task if the current state-of-the-art is maintained. Other Members suggested exploring the possibility of establishing ranges for groups or types of similar fruit juices instead of single values for individual juices, e.g. citrus fruit juices.

65. IFU recalled that the TFFJ concluded that establishing definitive quality and authenticity provisions was not feasible within its timeframe to finish the work assigned by CAC. The expert group would not attempt to develop numerical provisions for every quality and authenticity parameter but rather gather relevant information and demonstrate that the analytical methods in Annex 2 of CRD37 are appropriate for assessing the quality and authenticity of fruit juices and nectars. IFU highlighted that it was important to distinguish between measuring a value, which is what these methods are designed to do, and interpreting that value, which is more complex and critical challenge, and the most difficult aspect of the problem. The expert group could investigate the latter and report its findings to CCMAS45.

66. IFU confirmed that, due to the global nature of CXS 247-2005, establishing individual provisions for each juice would not be feasible, and that would be, in any case, the task for Codex and not for the expert group. IFU mentioned that for many of the analytical methods listed in Table 2, a normal range of values exists; however, it would not be possible to address the 70+ juice types covered by CXS 247-2005. Moreover, some parameters serve dual purposes, i.e. quality and authenticity, and labelling was insufficient to address issues associated with misleading practices. Because of these complexities, setting a single fixed value for specific provisions was not feasible. In some cases, it would be possible to define acceptable ranges; however, that would also vary across the 70+ juice types.

67. The Chairperson acknowledged that under the current situation of CXS 247-2005 and the procedures in place in the Procedural Manual for the endorsement of methods of analysis, a numerical value or range was necessary to determine whether the method is “fit-for-purpose”. However, it might be worthwhile exploring whether it would be possible to determine a range of the measurement or a maximum limit of detection (LOD) that provides a basis to assess the methods.

68. The Chairperson presented three options for consideration:

- **Option 1**: Revoke the methods at this Session or CCMAS45, due to the lack of clearly defined provisions or measurement ranges. Without this information, it is not possible to properly evaluate whether the methods are “fit-for-purpose”. This would effectively halt progress and undo substantial work already done.

- **Option 2**: Take no further action and wait for a future revision of CXS 247-2005. However, since there is no active commodity committee and no timeline for such a revision, CCMAS could wait indefinitely. This would effectively put the issue on hold without resolution.
- **Option 3**: Convene an expert group to begin work on a system of provisions for authenticity and quality. This group will report back to CCMAS45 with an update on the authenticity issue. If CCMAS finds the proposed solution is scientifically sound and procedurally appropriate, CCMAS45 could re-establish the EWG to review the methods in Annex 2 and those methods transferred from Annex 1.

69. The Chairperson further noted that, in practice, there were two more realistic paths forward: either take no action, which leaves the problem unresolved, or take proactive steps and begin the work with an expert group.

Conclusion

70. Based on the discussion and information provided, and the options available, CCMAS44 agreed to convene an expert group under the auspices of IFU with the following terms of reference:

i. The expert group will:

a. determine the ranges of parameters relevant for quality and authenticity;

b. determine the range of measurements or maximum LOD;

c. evaluate the present list of endorsed methods in CXS 234-1999 and CXS 247-2005;

d. determine/consider if the presently endorsed methods are still appropriate and "fit-for-purpose" to control the "quality and authenticity" of fruit juices;

e. determine/consider if any of the presently endorsed methods should be revoked and eliminated from CXS 234-1999 and CXS 247-2005;

f. assess if any new methods could be considered by the CCMAS endorsement working group in the future, for addition to CXS 234-1999 for the general provision of juice "quality and authenticity"; and

g. collate relevant validation data for any new procedures that CCMAS could consider in the future.

ii. The expert group will prepare a discussion paper for consideration by CCMAS45 on which methods in CXS 234-1999 and CXS 247-2005 are still considered important and "fit-for-purpose", in their expert opinion, and which methods should be maintained in CXS 234-1999, revoked, or replaced.

71. Codex Members interested in contributing to this work were invited to contact IFU and nominate fruit juice experts to participate in the group in their individual capacity.

COCOA PRODUCTS AND CHOCOLATE WORKABLE PACKAGE (Agenda item 5.2)[10]

72. Serbia, as Chair of the EWG and speaking also on behalf of the co-chair, USA, introduced the item. The EWG Chair recalled that although the number of methods under review had been limited, the technical issues had been complex. Several questions had remained open, including the use of non-selective methods for fat determination in cocoa products and the need for fit-for-purpose methods to quantify fat-free milk solids. It was suggested that the EWG should be re-established to continue to address these matters.

73. USA, as co-chair of both the EWG and the VWG, reported that during the VWG meeting, a number of recommendations had been agreed. The co-chair proposed advancing those provisions before the establishment of the EWG.

Discussion

74. CCMAS44 noted that while the VWG had addressed some provisions, others remained unconsidered. CCMAS44 considered all provisions in CRD02 Rev.1, Appendix V, and made the following additional comments and decisions.

Cocoa butter

75. CCMAS44 considered the provision for cocoa butter in chocolate products under the *Standard for chocolate and chocolate products* (CXS 87-1981) and noted the VWG's recommendation to determine fat using AOAC 963.15 / ICA No. 14 and moisture as water using AOAC 977.10 / ICA No. 26, based on method

[10] CL 2025/17-MAS; CX/MAS 25/44/7; CX/MAS 25/44/7-Add.1 (Comments of Australia, Egypt, Ghana, Iran (Islamic Republic of), Iraq, Peru, Thailand, United Kingdom (UK) and European Cocoa Association, ICUMSA, International Confectionery Association (ICA))

precision. It was clarified that AOAC 963.15 / ICA No. 14 did not include a moisture correction; consequently, AOAC 977.10 / ICA No. 26 remained necessary. CCMAS44 noted cocoa butter determined as fat was true for products without added other fats (e.g. milk fat), and that additional methods might be required for products containing such other added fats.

76. CCMAS44 agreed to refer this provision to the re-established EWG (hereafter referred to as the EWG) for further consideration.

Fat, total on dry basis

77. Noting that the VWG had recommended removing the provision, CCMAS44 agreed instead to retain it in CXS 234-1999, with amendments, as there was a provision for total fat in CXS 87-1981 (Section 2.1.6).

Milk fat

78. CCMAS44 noted that the proposed ICA No. 5 method was a very old method and might not be appropriate and agreed to refer this provision to the EWG for further consideration.

Moisture

79. The co-chair of the EWG and VWG explained that major changes were made to the methods currently listed in CXS 234-1999. It was explained that the two methods which were different methods were both listed as Type I, one being Gravimetry-drying at 100-102ºC and the other Titrimetry-Karl Fischer, which measures only water molecules. The VWG had recommended that the ICA No.1 be endorsed as Type IV and other methods as Type II. CCMAS44, however, noted that further discussion might be needed on the co-existence of a Type II rational method and a Type IV defining method and referred this provision to the EWG for further consideration.

Non-cocoa butter vegetable fat

80. CCMAS44 noted that the VWG had recommended endorsement of AOCS Ce 10-02 as a Type IV method for non-cocoa butter vegetable fat and clarified that the method should be described as “GC-MS.” CCMAS44 further noted that ISO methods for detecting cocoa butter equivalents in chocolate could be Type I methods, potentially conflicting with the proposed Type IV classification.

81. In light of this, CCMAS44 agreed to refer this provision to the EWG for further consideration.

Cocoa shell

82. CCMAS44 noted that AOAC 968.10 and AOAC 970.23, previously combined in CXS 234-1999 under a single provision, were distinct methods with different analytes. The VWG had recommended splitting the provision into two separate lines to enhance clarity. The revised provision names were “Cocoa shell – determined as spiral vessel” and “Cocoa shell – determined as stone cell count,” with both methods classified as Type I.

83. CCMAS44 agreed with this approach.

Fat-free cocoa solids, fat-free milk solids

84. CCMAS44 agreed with the VWG’s recommendation to endorse the revisions to these two provisions.

Other provisions

85. CCMAS44 agreed to refer the remaining provisions to the EWG for further consideration.

Conclusion

86. CCMAS44 agreed to:

i. submit the methods for adoption/revocation by CAC48 (Appendix II, Part 1.1); and

ii. re-establish the EWG chaired by Serbia and co-chaired by USA, working in English to:

a. continue reviewing the relevant methods in the cocoa products and chocolate workable package (Appendix II, Part 3.1); and

b. prepare and submit the report of the EWG to the Codex Secretariat at least three months prior to CCMAS45.

Other matters

87. CCMAS44 considered review of additional workable packages and agreed to:

i. start the review of methods in the sugars and honey workable package; and

ii. establish an EWG chaired by Uruguay, working in English and Spanish, to:

a. review the sugars and honey workable package; and

b. prepare and submit the report of the EWG to the Codex Secretariat at least three months prior to CCMAS45.

SAMPLING PLANS (Agenda item 6)

INFORMATION DOCUMENT: *GENERAL GUIDELINES ON SAMPLING* (CXG 50-2004) - E-BOOK WITH SAMPLING PLANS APPLICATIONS (Agenda item 6.1)[11]

88. New Zealand, as Chair of the EWG, speaking also on behalf of the co-chair, Germany, introduced the item and recalled that the information document would support the implementation of the revised *General guidelines on sampling* (CXG 50-2004). The EWG Chair informed CCMAS44 of the background of the development of the information document; the work process followed by the EWG to develop the information document; its purpose, structure, and content including applications (Apps); conclusions and recommendations for consideration by CCMAS.

89. The EWG Chair recalled that the information document was not an official Codex text as it is neither developed through the Codex Step Procedure nor adopted by CAC. However, it would be available for internal use by CCMAS and Codex committees and for public consultation via the Codex website[12] following the Committee's agreement. The information document was considered a living document, meaning it could be revised by CCMAS as needed.

90. The EWG Chair explained that the document's purpose is to provide further information on the sampling plans referred to in CXG 50-2004, examples for each of the main types of sampling plans, additional information on other sampling plans, including Bayesian plans, and links to the apps for the design and evaluation of these sampling plans.

91. The EWG Chair explained that App 1 evaluates and designs sampling plans for homogeneous lots, which is included in the information document by link. Apps 2 and 3 were being developed and would be included in the information document once available. As more apps were developed, links to these apps would be included in the information document and available in English only. The EWG Chair noted that other resources (e.g. video clips, webinars) would be provided to the extent possible on the Codex website to support the implementation and understanding of the sampling plans provided by these apps. In this regard, the EWG Chair referred to the webinar on 28 April 2025 to provide information on sampling plans in CXG 50-2004 and other types of sampling plans (including Bayesian plans) and to demonstrate App 1 based on Codex provisions.

92. The EWG Chair further explained the structure and content of the information document. She recalled that delegations at CCMAS42 (2023) and CCMAS43 (2024) supported the inclusion of sampling plans employing smaller sample sizes or less testing, therefore, some information on Bayesian plans was included. Following written comments submitted to this Session, the information document was restructured to include a new Part 3 to cover Bayesian plans based on risk- or utility-based approaches, while the overall content remained unchanged.

93. Based on the above information, CCMAS agreed to consider the revised information document (CRD35) and noted general support for it and for its publication on the Codex website. However, as summarized below, CCMAS44 noted comments and clarifications regarding the inclusion of Bayesian plans.

94. One Member did not agree to introduce a new concept of Bayesian plans into the information document and noted that CAC37 (2014) endorsed that information documents should be by-products of ongoing work of the Committee, which is the review of CXG 50-2004 in this case. The scope of CXG 50-2004 explicitly indicated that the focus is on acceptance sampling plans for inspecting isolated homogeneous lots. The Bayesian sampling plan applied to continuous lots but was unsuitable for isolated ones. If CCMAS was interested in considering Bayesian sampling plans, it should be done by elaborating a Codex text rather than an information document. Thus, the Member proposed deleting all Bayesian plan references throughout the information document.

95. The EWG Chair clarified that delegations at previous sessions of CCMAS had shown considerable interest in Bayesian sampling plans and strongly supported their inclusion in the information document. These plans provided a potential way to reduce testing costs. The Bayesian sampling approach was an area of international scientific work, including within ISO, which would soon publish a technical report on applying Bayesian methods to acceptance sampling. Therefore, it was foreseeable that new work might also start in CCMAS through the Codex Step Procedure. The EWG Chair recalled that information documents were in nature, not official Codex texts (see paragraph 89) and mentioned that:

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11 CL 2025/18-MAS; CX/MAS 25/44/8; CX/MAS 25/44/8-Add.1 (Comments of Colombia, Egypt, Indonesia, Japan, Kenya, Norway, Philippines and Thailand)

12 https://www.fao.org/fao-who-codexalimentarius/resources/inf-doc/en/

- as an initial step, Bayesian sampling plans were included in the information document and separated from the other classic sampling plans in CXG 50-2004, which are presented in Parts 1 and 2 of the document; and
- by providing a new Part 3, an overview of Bayesian sampling plans could be provided, that are the subject of new and ongoing work in ISO and other forums.

96. In the spirit of compromise, the Member agreed to the publication of the information document on the Codex website. Other Members supported the explanation of the EWG Chair and thus the inclusion of Bayesian sampling plans in the information document to facilitate their understanding.

97. Another Member, while supporting the inclusion of Bayesian sampling plans in the information document, indicated that it would be necessary to complement the information given in the information document with that contained in the *Principles for the use of sampling and testing in international food trade* (CXG 83-2013). It was recalled that the information document was a living document that could continually be improved at future sessions of CCMAS (see paragraph 89) and that this proposal could be considered as part of the future updates to the information document.

98. CCMAS44 noted that there was an existing information document on practical examples of sampling currently available on the Codex website and considered whether to remove the document in light of the agreement to publish the information document (e-book with sampling plan applications) or whether the two information documents should co-exist on the Codex website.

99. The EWG Chair confirmed that the information document on practical examples of sampling should be removed from the website as it was based on a particular set of assumptions that may not be fulfilled in practice; however, the EWG would review this document, particularly about the format of how sampling plans can be consistently presented in CXS 234-1999 or a separate Codex standard on sampling plans (see Agenda item 6.2, review of sampling plans).

Conclusion

100. CCMAS44:

i. agreed to publish the information document as revised (Appendix IV) and to inform Codex committees of the publication of this document;

ii. agreed to remove the current information document titled “Practical examples of sampling plans” from the Codex website;

iii. noted that as other Apps are being developed, they would be forwarded to the Codex Secretariat for inclusion to the list of Apps in the information document and that CCMAS would be informed accordingly; and

iv. noted that other supporting resources, such as webinars, would be made available on the CCMAS webpage.

REVIEW OF SAMPLING PLANS IN CXS 234 (Agenda item 6.2)[13]

101. New Zealand, as Chair of the EWG, speaking also on behalf of the co-chair, Germany introduced the item and explained that the EWG considered two matters, namely, the review of sampling plans in CXS 234-1999 and a proposal to develop a discussion paper on sampling plans for bulk material, including mycotoxins.

Review of sampling plans in CXS 234-1999

102. The EWG Chair recalled that the purpose of the discussion paper was to provide a review of current procedures for including sampling plans in CXS 234-1999 and to consider sampling plan information that may be included in CXS 234-1999 for sampling plans that will be developed under CXG 50-2004, including sampling plans from other sources subject to endorsement by CCMAS.

103. The EWG Chair summarized the approaches that could be taken by CCMAS in considering sampling plans, namely:

- Include sampling plan information in CXS 234-1999 while noting that a review of the current format is needed.
- Include sampling plan information in each individual Codex standard.

[13] CX/MAS 25/44/9

- Develop a new standard to include sampling plan information (in parallel to CXS 234-1999) that only contains sampling plan information and remove Part B of CXS 234-1999.
- Develop a standard for each commodity group that describes the sampling plans for that commodity group.

104. The EWG Chair also summarized information that should be consistently provided on each sampling plan, regardless of the approach that CCMAS may take on the inclusion and maintenance of sampling plans.

105. The EWG Chair emphasized that CCMAS did not need to decide at the current session on the options and the format and content of the presentation of sampling plans, but that it would be helpful if delegates provided their views on these matters to continue progressing work in the EWG for consideration by CCMAS45.

106. CCMAS44 noted general support for re-establishing the EWG to continue investigating the issues raised in paragraphs 103-104 and the discussion paper. Furthermore, CCMAS44 noted the following comments and clarifications regarding the approaches for the location of sampling plans and the format and related information that should accompany their presentation.

Discussion

107. On the possible approaches that CCMAS may take regarding the location sampling plans, the Codex Secretariat proposed that the EWG continue discussing and carefully examine the different options given in paragraph 103, in particular the pros and cons of keeping the sampling plans in CXS 234-1999 as opposed to separating them into a new standard or retaining or including them in individual commodity standards.

108. Members generally favoured keeping all sampling plans in one place, either in CXS 234-1999 or in a separate standard, and presented in a consistent way. This would avoid inconsistencies and ensure the same approach to methods of analysis is also taken for sampling plans. This could also facilitate the digitalization of information in the future, vis-à-vis the ongoing considerations on developing a database for methods of analysis and sampling.

109. A Member noted that, although there might be a preference for keeping all sampling plans in one place, this might favour what is easiest for CCMAS rather than what best serves the broader Codex community. While CCMAS might prefer consolidating sampling plans into a single place for simplicity, commodity committees might prefer having those plans embedded directly within their own standards, so all the information related to the commodity is in the corresponding standard. Therefore, a discussion on the approaches should consider the different perspectives of Codex subsidiary bodies, including CCMAS, that may best suit Codex.

110. A Member Organization noted that while it was important to determine the approach to placing and displaying sampling plans, and that all approaches stated in paragraph 103 were still open for discussion, the EWG's initial analysis revealed that approximately two-thirds of the commodity provisions in CXS 234-1999 lacked sampling information, an issue that needed attention. Emphasis should therefore be placed on defining the *function* before determining the *form*. Consequently, the approach should be driven by practical needs, i.e. identifying appropriate sampling plans based on specific commodity-provision combinations, rather than simply fitting them into a predetermined document structure.

111. On the format and content of the information that should be displayed for a consistent presentation of sampling plans, there were questions on who should be responsible for providing information to generate the sampling plans (e.g. consumer's risk quality level, producer's risk quality level), either by CCMAS or commodity committees, etc. It was also noted that several commodity committees were adjourned *sine die* and that the EWG should examine the issue of who will be responsible for specifying sampling plans, especially in the case of active committees. A Member Organization expressed the view that such responsibility rests with the relevant committee when it exists.

112. The EWG Chair noted that the issue of who was responsible for specifying parameters such as acceptable quality limits, risk levels, or any other key parameters that determine the sampling plan to be applied should be further discussed in the EWG.

Development of sampling plans for lots consisting of bulk material/heterogenous lots, including for mycotoxins

113. The EWG Chair provided background on this proposal. The EWG Chair recalled that delegates at previous sessions of CCMAS had expressed considerable interest in acceptance sampling plans for lots consisting of bulk materials/heterogeneous lots, along with a corresponding app. During the work on CXG 50-2004 and the associated information document, there was an interest in reviewing the current sampling approaches used, such as the sampling plans for mycotoxins in the *General standard for contaminants and toxins in food and feed* (CXS 193-1995), and consideration of other approaches for sampling plans for these commodities. If a

proposal for acceptance sampling plans for bulk materials/heterogenous lot, including plans for mycotoxins is put forward, the Codex Committee on Contaminants in Foods (CCCF) would be consulted.

114. The EWG Chair explained that the review should address, but not be limited to the following issues:

- The number of increments in CXS 193-1995 (e.g. 100 increments) may lead to pockets of possibly harmful contamination being missed.
- Usually, a single composite sample is tested, meaning that the contamination levels within the lot are averaged out. The final result may be less than the acceptance limit, and possibly below the detection limit of the test method, even if potentially harmful pockets of contamination are present in the lot.
- Bayesian approaches may be more appropriate than risk-based approaches, so a method for calculating various (Bayesian) risks may be required.

115. The EWG Chair noted that gathering views from delegations on this proposal, and, if it were supported, would help guide further work in the EWG.

Discussion

116. CCMAS44 noted general support for the proposal, provided that CCMAS and CCCF collaborate closely in developing these sampling plans and it was confirmed that this would be the case.

117. The Codex Secretariat also explained that CCCF would be informed about the decisions and ongoing discussions of CCMAS at its upcoming session (June 2025). The Codex Secretariat further informed CCMAS that CCCF had planned to review sampling plans in CXS 193-1995 following the recommendations of CCMAS42, but this was put on hold to await the outcomes of the discussions on sampling plans in CCMAS.

Overall conclusion

118. CCMAS44:

i. agreed to continue developing the discussion papers on:

a. the review of sampling plans in CXS 234-1999, particularly the different approaches to placing the sampling plans in the standard(s), the format and content of the presentation of sampling plans, and the responsibility for assessing the parameters that determine the selection of the appropriate sampling plan for a given commodity/provision combination; and

b. the development of sampling plans for bulk materials/heterogenous lots, including mycotoxins, including proposed sampling plans for consideration by CCMAS45, and to inform CCCF of this decision.

ii. noted that work on sampling plans for bulk materials/heterogenous lots, including for mycotoxins, should be conducted in close collaboration with CCCF; and

iii. noted the possible need for CCMAS to provide support to commodity committees in their review of sampling plans.

119. CCMAS44 further agreed to re-establish the EWG, chaired by New Zealand and co-chaired by Germany, working in English only, to:

i. carry out the work described in paragraph 118; and

ii. prepare a discussion paper relating to sampling plans for bulk materials/heterogenous lots, including mycotoxins, for presentation to CCMAS45.

120. The report of the EWG should be made available to the Codex Secretariat at least three months prior to CCMAS45.

NUMERIC PERFORMANCE CRITERIA FOR THE DETERMINATION OF NITRATE AND NITRITE IONS IN FOOD MATRICES (Agenda Item 7)[14]

121. Australia, as Chair of both the EWG and the VWG, speaking also on behalf of the co-chair, USA, introduced the item. The Chair recalled that CCFA had requested CCMAS to: (i) establish NPC for methods of analysis in specified food matrices; (ii) provide information on available analytical methods suitable for both the adopted MLs and the lowest proposed residue levels; and (iii) clarify whether the methods measured nitrate and nitrite ions separately or in combination.

[14] CX/MAS 25/44/10

122. The Chair reported that the EWG had:

- developed NPC for both the adopted MLs and the lowest proposed residue levels in the relevant food matrices; and
- reviewed the list of methods submitted by CCFA including one additional recently published method and provided a summary of the method validation data in Appendix 3 of CX/MAS 25/44/10 for assessment against the NPC.

123. "Examples of applicable methods that met the established criteria" that meet the NPC had been identified from Appendix 3 and were presented in Appendices 1 and 2 of CX/MAS 25/44/10.

124. The Chair further explained that the VWG had reviewed Appendices 1 and 2 of CX/MAS 25/44/10 and made minor editorial revisions. The revised versions were presented in CRD02 Rev.1, Appendix VI. It was highlighted that, should CCFA decide to revise the lowest proposed residue levels, suitable analytical methods could be selected based on the information provided in Appendix 3 of CX/MAS 25/44/10.

Conclusion

125. CCMAS44:

i. agreed with the NPC for nitrate and nitrite in specified food matrices, as presented in Appendix V; and

ii. agreed to forward the abovementioned NPC, together with the remaining information in CX/MAS 25/44/10, including Appendix III in this document.

METHODS OF ANALYSIS FOR PRECAUTIONARY ALLERGEN LABELLING (Agenda Item 8)[15]

126. USA, as Chair of the EWG, speaking also on behalf of the co-chair, the United Kingdom (UK), introduced the item. The Chair reported that the EWG report had been considered during the VWG meeting, and there had been general agreement that the output of the EWG was not suitable for referral to CCFL, due to the late availability of the report. A proposal to re-establish the EWG to adequately address the request from CCFL was noted during the VWG discussions.

Conclusion

127. CCMAS44 agreed to re-establish an EWG chaired by USA and co-chaired by UK, working in English to:

i. finalize review of the methods in CX/MAS 25/44/11 against the available validation guidelines and performance requirements[16,17];

ii. simplify the presentation of methods and their validation status included in CX/MAS 25/44/11 Appendix II;

iii. develop a draft response for consideration by CCMAS45 to CCFL49; and

iv. prepare and submit the report of the EWG to the Codex Secretariat at least three months before CCMAS45.

128. CCMAS44 confirmed that the EWG would not address the second question from CCFL regarding sampling plans.

HARMONIZATION OF NAMES AND FORMAT FOR PRINCIPLES IDENTIFIED IN CXS 234 (Agenda Item 9)[18]

129. Brazil, as Chair of the EWG and speaking also on behalf of the co-chair, Chile, introduced this item. Brazil recalled earlier decisions of CCMAS which were to: (i) establish a centralized database consolidating all methods relevant to CCMAS; (ii) harmonize terminology for analytical methods across Codex standards; and (iii) develop a publicly accessible database on methods of analysis and sampling for inclusion on the Codex website. The work of the EWG to develop harmonized terminology for analytical methods, format for principles and provision names was thus to support the development of the database.

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15 CX/MAS 25/44/11

16 Dr. Latimer, George W, Jr. (ed.), 'Validation Procedures for Quantitative Food Allergen ELISA Methods: Community Guidance and Best Practices', in Dr. George W Latimer, Jr. (ed.), Official Methods of Analysis of AOAC INTERNATIONAL, 22nd Edition (New York, 2023; online, AOAC Publications, 4 Jan. 2023)

17 EN 17855:2024(Main) Foodstuffs - Minimum performance requirements for quantitative measurement of the food allergens milk, egg, peanut, hazelnut, almond, walnut, cashew, pecan nut, Brazil nut, pistachio nut, macadamia nut, wheat, lupine, sesame, mustard, soy, celery, fish, molluscs and crustaceans

18 CX/MAS 25/44/12

130. Brazil recalled the activities undertaken previously and discussions held within the EWG established by CCMAS43. Based on these consultations, a discussion paper was developed, comprising one appendix and four annexes as outlined below:

- Appendix I: Discussion paper on harmonization of names and format for principles and provisions in CXS 234-1999
- Annex A: Principles of methods of analysis
- Annex B: Acronyms and abbreviations of principles of methods of analysis
- Annex C: List of acronyms for standard method references
- Annex D: List of provisions

131. Brazil summarized the EWG discussions, key comments received and their corresponding responses. Regarding a suggestion to amend the first paragraph of Section 1 – General Guideline in Appendix I to specify techniques for sample preparation, extraction, and separation when critical to the determination process, Brazil clarified that only techniques directly related to obtaining the test result should be reflected in the principle name, as sample preparation steps were already detailed within the methods themselves. This suggestion was therefore not accepted. Comments on excluding Annex D were also noted, with concerns raised that such content had already been addressed in previous revisions and should remain part of future updates.

132. Brazil proposed publishing Appendix I along with annexes A, B, and C as an information document to support understanding and facilitate access to relevant information. It was further emphasized that the information document should be a living document and would be updated as needed. Regarding Annex D, Brazil recommended further discussion at CCMAS45, emphasizing the need to consult with the relevant Codex Committees to accurately reflect provision names from the commodity standards.

Discussion

133. Members expressed appreciation for the preparation of this information document, recognizing its importance and broad applicability. There was general support for its inclusion under the information document "Comprehensive Guidance for the Process of Submission, Consideration, and Endorsement of Methods for Inclusion in CXS 234".

134. CCMAS44 reviewed Appendix I and annexes A, B, C, and D, noting the decision to consider Annex D separately due to its complexity. In addition to editorial corrections, CCMAS44 made the following comments and decisions:

<u>Appendix I: Discussion paper on harmonization of names and format for principles and provisions in CXS 234-1999</u>

135. CCMAS44 agreed to:

- revise the title of the Appendix to "Harmonization of names and format for principles and provisions in CXS 234-1999";
- delete the second paragraph under Section 1 – General Principle, as Annex D would be considered separately;
- include definitions for "Potentiometry", "Spectroscopy", and "Mass Spectrometry (MS)", and revise the definitions for "Biological Assay", "Chromatography", "Sensory Assay" and "visual examination": under Section 2;
- delete "Type I Methods" from the header of Section 3.1, and amend terminology within the section by replacing "humidity" with "moisture", "strange matters" with "foreign matter", and "lipids" with "fat"; and
- delete "Type II Methods" from the header of Section 3.2.

136. Changes to the headers of sections 3.1 and 3.2 was to keep the headers general as the intent of the sections are to show how to mention principles for the two kinds of methods and not to define the type of methods that are already provided for in the Procedural Manual.

137. An Observer, speaking on behalf of the Inter-Agency Meeting (IAM) on methods of analysis, expressed concern about potential duplication with the existing information document titled "Comprehensive Guidance for the Process of Submission, Consideration, and Endorsement of Methods for Inclusion in CXS 234" and suggested that a more comprehensive review might be beneficial to ensure consistency and avoid redundancy.

Annex A: Principles of methods of analysis

138. CCMAS44 agreed to:

- move “Enzyme Linked Immunosorbent Assay (ELISA)” under “Immunoassay”;
- move “Electrometric” under “Electrophotometry”;
- include “microwave oven drying” under “Gravimetry”;
- include “Quadrupole Time-of-Flight (QTOF)” under “Gas Chromatography (GC)”;
- delete the specific temperatures for “ashing” and “drying”, and express them as “Ashing at (temperature) °C” and “Drying at (temperature) °C”; and
- delete the word “Thermal” from “Thermal Conductivity Detector (TCD)” and consequentially amend the acronym “TCD” to “CD”.

139. A Member also suggested that the example of QTOF could be generalised to “High resolution detectors”.

140. CCMAS44 noted that some principles included in CXS 234-1999 were missing from the Annex, while others listed in the Annex were not present in CXS 234-1999. CCMAS44 also noted inconsistencies in terminology (e.g. "Electrophotometry" vs "Photometry", "Electrometric") and suggested including examples to illustrate the correct presentation of principles.

141. CCMAS44 agreed that this Annex required further review and was not yet ready for publication.

Annex C: List of acronyms for standard method references

142. CCMAS44 noted that “IOCCC” should be replaced with “ICA” and corrected the full name of “FDA” to “Food and Drug Administration”.

Annex D: List of provisions

143. CCMAS44 noted the view that the provisions in CXS 234-1999 should remain aligned with those in the commodity standards to avoid misalignment and confusion. It was further emphasized that the provisions in CXS 234-1999 reflected the original commodity standard provisions and should not be oversimplified as this could remove essential context. Some provisions indicated method complementarity and had been the subject of extensive discussions. Some of the proposed changes in Annex D carried significant implications. Therefore, consultation with commodity committees and other relevant committees was necessary.

144. The Chairperson proposed separating the provisions into three groups: those requiring no changes or only editorial changes; those linked to active commodity committees; and those related to commodity committees that had been adjourned *sine die* or abolished. Appropriate actions, including seeking guidance from the respective committees on changes to these provisions, would then be considered.

145. In response to whether CCMAS could change provision names in commodity standards for Codex Committees that had been adjourned *sine die*, the Codex Secretariat clarified that, in general, CCMAS did not have the authority to make such changes. If necessary, such proposals should be referred to the CAC. A similar situation occurred with revisions to the provision in one milk and milk products standard (i.e. amendment of the provision in section 3.3 of the *Standard for edible casein products* (CXS 290-1995))[19], which followed the same mechanism.

146. CCMAS44 agreed to further consider this topic at the next session.

Conclusion

147. CCMAS44 agreed to re-establish the EWG, chaired by Brazil and co-chaired by Chile, working in English, to:

i. further revise the “Harmonization of Names for Principles in CXS 234-1999”, including annexes A, B, and C, using Appendix VI as the basis, with the aim of ensuring that the principles in CXS 234-1999 are properly included;

ii. continue discussions on Annex D, focusing on separating the provisions into three distinct groups (editorial or no-change provisions, provisions linked to active committees, provisions linked to inactive committees) and making corresponding recommendations; and

iii. prepare and submit the report of the EWG to the Codex Secretariat at least three months before CCMAS45.

[19] REP21/MAS paragraphs 23 and 24(ii)

148. CCMAS44 further agreed that following agreement of the revisions at a future session, the document "Harmonization of Names for Principles in CXS 234-1999," along with annexes A, B, and C, would be introduced under Section 4.0 (i), in the information document titled "Comprehensive Guidance for the Process of Submission, Consideration, and Endorsement of Methods for Inclusion in CXS 234" and incorporated into the information document as an Annex.

REPORT OF AN INTER-AGENCY MEETING ON METHODS OF ANALYSIS (Agenda item 10)

149. The Observer from the United States Pharmacopeial Convention (USPC), speaking as Chair of the IAM, introduced the IAM report described in CRD03, and asked participants to contact IAM or SDOs directly if there were concerns. The Observer highlighted several recurring topics from the current session that should warrant future discussion, which were:

- concerns over potential expansion beyond what was originally intended in the use of Type I methods, particularly regarding cereals and cocoa products, where different methods are applied to the same provision;
- method retention and withdrawal, noting different revision cycles across organizations and suggesting a structured period for method replacement within CXS 234-1999 requires careful consideration since analysts are always expected to use the latest version of official methods;
- the need to revisit the historical discussion of matrix extension of official methods, and the longstanding, associated challenges of providing precision data; and
- ongoing difficulties related to authenticity parameters, especially in the standards for fruit juices, vegetable oil, and olive oil.

150. CCMAS44 noted that several of the issues raised in CRD03 had been considered under relevant agenda items.

Conclusion

151. CCMAS44 thanked the members of IAM for their valuable contribution to the work of CCMAS44.

OTHER BUSINESS AND FUTURE WORK (Agenda item 11)

152. CCMAS44 noted that no other business and future work had been proposed.

DATE AND PLACE OF NEXT SESSION (Agenda item 12)

153. CCMAS44 was informed that its 45th Session was tentatively scheduled to take place from 9-13 March 2026 in Budapest, Hungary, with the final arrangements subject to confirmation by the Host Country in consultation with 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 AND REVOCATION BY CAC48

1.1 COCOA PRODUCTS AND CHOCOLATE

1.2 FISH AND FISHERY PRODUCTS

1.3 FOODS FOR SPECIAL DIETARY USES

1.4 FRUIT JUICES AND NECTARS

1.5 MILK AND MILK PRODUCTS

1.6 MISCELLANEOUS PRODUCTS

Part 2. METHODS OF ANALYSIS WHICH REMAIN UNCHANGED IN CXS 234 AS A RESULT OF DECISIONS BY CCMAS44

2.1 CEREALS, PULSES AND LEGUMES AND DERIVED PRODUCTS

2.2 FRUIT JUICES AND NECTARS

2.3 MILK AND MILK PRODUCTS

2.4 MISCELLANEOUS PRODUCTS

Part 3. METHODS OF ANALYSIS FOR FURTHER CONSIDERATION

3.1 COCOA PRODUCTS AND CHOCOLATE

3.2 FOODS FOR SPECIAL DIETARY USES

3.3 FRUIT JUICES AND NECTARS

Part 1

METHODS OF ANALYSIS AND SAMPLING FOR ADOPTION AND REVOCATION BY CAC48

Notes:

1. Methods and performance criteria for inclusion and/or amendment in CXS 234-1999: changes indicated in ~~strikethrough~~, or **bold** and underlined font.
2. Methods for revocation in CXS 234-1999: ~~strikethroughs~~ are indicated in red.
3. The references to Appendix VIII and Appendix XI in this document relate to the relevant appendices in CXS 234-1999.

1.1 COCOA PRODUCTS AND CHOCOLATE

Cocoa products and chocolate				
Commodity	**Provision**	**Method**	**Principle**	**Type**
Chocolate and chocolate products	Fat-free cocoa solids	**ICA No. 26 / AOAC 977.10 and** AOAC 931.05	~~Oven evaporation and factor~~ **Calculation from moisture (Determined as water) and gravimetry**	I
Chocolate and chocolate products	Fat-free milk solids **(Determined as Milk Protein)**	**ICA No. 26 and ICA No. ~~IOCCC~~ 17 and ICA No. 13 ~~or~~ / AOAC 977.10 and AOAC 955.04C and** AOAC 939.02	**Calculation from moisture content, and** Titrimetry~~,~~ **(**Kjeldahl digestion**)~~;~~ content of extracted and precipitated** ~~after extraction of~~ milk protein~~s~~.	I
Chocolate and chocolate products	Fat, total **on dry basis**	**ICA No. 26 / AOAC 977.10 and** AOAC 963.15	**Calculation from moisture (Determined as Water) and** Gravimetry (Soxhlet extraction)	I
Cocoa (cacao) mass or cocoa/ chocolate liquor, and cocoa cake	Cocoa shell **(determined as spiral vessel count)**	AOAC 968.10	**Microscopy -** Spiral vessel count~~, stone cell count~~	I
Cocoa (cacao) mass or cocoa/ chocolate liquor, and cocoa cake	Cocoa shell **(determined as stone cell count)**	AOAC 970.23	**Microscopy -** ~~Spiral vessel count,~~ stone cell count	I

1.2 FISH AND FISHERY PRODUCTS

Fish and fishery products				
Commodity	**Provision**	**Method**	**Principle**	**Type**
Fish sauce	Amino acid nitrogen	AOAC 920.04 and AOAC 920.03	Determining formaldehyde titration method **and** Subtracting by ammoniacal nitrogen (magnesium oxide method)	I
Quick frozen fish sticks (fish fingers), fish portions and fish fillets – breaded or in batter	**Determination of fish content (declaration) – Nitrogen**	**ISO 937**	**Titrimetry (Kjeldahl digestion)**	**II**
Quick frozen fish sticks (fish fingers), fish portions and fish fillets – breaded or in batter	**Determination of fish content (declaration) – Moisture**	**ISO 1442**	**Gravimetry**	**I**
Quick frozen fish sticks (fish fingers), fish portions and fish fillets – breaded or in batter	**Determination of fish content (declaration) – Total fat**	**ISO 1443**	**Gravimetry**	**I**
Quick frozen fish sticks (fish fingers), fish portions and fish fillets – breaded or in batter	**Determination of fish content (declaration) – Ash**	**ISO 936**	**Gravimetry**	**I**
~~**Salted Atlantic herring and salted sprat and sturgeon caviar**~~	~~**Determination of salt content**~~	~~**See Appendix VIII**~~		
~~**Salted fish and dried salted fish of the Gadidae family of fishes**~~	~~**Salt saturation**~~	~~**See equation in footnote**[xii]~~	~~**Calculation**~~	~~**I**~~

[xii] ~~The % salt saturation is calculated as follows:~~

~~1. % salt in water = (% salt content / (% salt content + % moisture)) x 100%~~

~~2. % salt saturation = (% salt in water / 26.4 %*) x 100%~~

~~* The solubility of sodium chloride in water is 36 g per 100 g water, and the constant is calculated as follows: 36 g sodium chloride / (100 g water + 36 g sodium chloride) x 100% = 26.4%~~

APPENDIX VIII

~~PREPARATION OF FISH SAMPLES AND~~ DETERMINATION OF ~~SALT AND~~ WATER CONTENT IN FISH AND FISHERY PRODUCTS

~~PART 1: PREPARATION OF FISH SAMPLES~~

~~Salted fish and dried salted fish of the Gadidae family of fishes~~

~~1. Before preparing of a subsample adhering salt crystals should be removed by brushing from the surface of the sample without using water.~~

~~2. The preparation of fish samples for the determination of salt content, and water content in order to calculate the % salt saturation of the fish should be carried out according to AOAC 937.07. The analysis should be on the edible portion of the fish.~~

~~3. Determination should be performed at least in duplicate.~~

~~PART 2: DETERMINATION OF SALT CONTENT~~

~~Salted fish and dried salted fish of the Gadidae family of fishes, salted Atlantic herring and salted sprat, and sturgeon caviar~~

~~1. Principle~~

~~The salt is extracted by water from the pre-weighed sample. After the precipitation of the proteins, the chloride concentration is determined by titration of an aliquot of the solution with a standardized silver nitrate solution (Mohr method) and calculated as sodium chloride.~~

~~2. Equipment and chemicals~~

- ~~Brush~~
- ~~Sharp knife or saw~~
- ~~Balance, accurate to □ 0.01 g~~
- ~~Calibrated volumetric flasks, 250 ml~~
- ~~Erlenmeyer flasks~~
- ~~Electric homogenizer~~
- ~~Magnetic stirrer~~
- ~~Folded paper filter, quick running~~
- ~~Pipettes~~
- ~~Funnel~~
- ~~Burette~~
- ~~Potassium hexacyano ferrate (II), K4Fe(CN)6·3H2O, 15% w/v (aq)~~
- ~~Zinc sulphate, ZnSO4·6H2O, 30% w/v (aq)~~
- ~~Sodium hydroxide, NaOH, 0.1 N, 0.41% w/v (aq)~~
- ~~Silver nitrate, AgNO3, 0.1 N, 1.6987% w/v (aq), standardized~~
- ~~Potassium chromate, K2CrO4 5% w/v (aq)~~
- ~~Phenolphthalein, 1% in ethanol~~
- ~~Distilled or deionized water~~

~~3. Procedure~~

~~(i) Five grams of homogenized subsample is weighted into a 250 ml volumetric flask and vigorously shaken with approximately 100 ml water.~~

~~(ii) Five millilitres of potassium hexacyano-ferrate solution and 5 ml of zinc sulphate solution are added, the flask is shaken.~~

~~(iii) Water is added to the graduation mark.~~

~~(iv) After shaking again and allowing to stand for precipitation, the flask content is filtered through~~

~~a folded paper filter.~~

~~(v) An aliquot of the clear filtrate is transferred into an Erlenmeyer flask and two drops of phenolphthalein are added. Sodium hydroxide is added dropwise until the aliquot takes on a faint red colour. The aliquot then diluted with water to approximately 100 ml.~~

~~(vi) After addition of approximately 1 ml potassium chromate solution, the diluted aliquot is titrated under constant stirring, with silver nitrate solution. End-point is indicated by a faint, but distinct, change in colour. This faint reddish-brown colour should persist after brisk shaking.~~

~~To recognize the colour change, it is advisable to carry out the titration against a white background.~~

~~(vii) Blank titration of reagents used should be done.~~

~~(viii) End-point determination can also be made by using instruments like potentiometer or colorimeter.~~

~~4. Calculation of results~~

~~In the equation of the calculation of results the following symbols are used:~~

~~A= volume of aliquot (ml)~~

~~C= concentration of silver nitrate solution in N~~

~~V= volume of silver nitrate solution in ml used to reach end-point and corrected for blank value~~

~~W= sample weight (g)~~

~~The salt content in the sample is calculated by using the equation:~~

~~Salt concentration (%) = (V x C x 58.45 x 250 x 100) / (A x W x 1000)~~

~~Results should be reported with one figure after the decimal point.~~

~~5. Reference method~~

~~As reference method a method should be used which includes the complete ashing of the sample in a muffle furnace at 550 °C before chloride determination according to the method described above (leaving out steps (ii) and (iv)).~~

~~6. Comments~~

~~By using the given equation all chloride determined is calculated as sodium chloride. However it is impossible to estimate sodium by this methodology, because other chlorides of the alkali and earth alkali elements are present which form the counterparts of chlorides.~~

~~The presence of natural halogens other than chloride in fish and salt is negligible.~~

~~A step, in which proteins are precipitated (ii), is essential to avoid misleading results.~~

~~PART 3: DETERMINATION OF WATER CONTENT~~

Salted fish and dried salted fish of the *Gadidae* family of fishes

i) Determination of % salt saturation as required by the standard, should be in accordance to AOAC 950.46.B (air-drying (a)).

ii) Determination of water content in the whole fish, when needed in the commercial trade of klippfish and wet salted fish, the method of sampling the fish should be carried out according to the “Determination of water content in whole fish by cross section method” defined in the annex to this appendix.

Salted Atlantic herring and salted sprat

Determination of water content is performed according to AOAC 950.46B (air-drying).

Table 1. Method performance criteria for sodium chloride and for salt determined as chloride expressed as sodium chloride

Commodity	Provision	ML (%)	Min. appl. range (%)	LOD (%)	LOQ (%)	Precision (RSD_R) (%) no more than	Recovery (%)	Examples of applicable methods that meet the criteria	Principle
Boiled dried salted anchovies	Sodium chloride and salt determined as chloride expressed as sodium chloride	15 (NaCl) 9.1 (Cl^-)	13.8–16.2 8.3–9.9	1.5 0.91	3.0 1.8	5.3 5.7	98–102 98–102	NMKL 178 AOAC 971.27 AOAC 937.09	~~Potentiometric titration~~ Titrimetry (Potentiometric) ~~Potentiometric titration~~ Titrimetry (Potentiometric) ~~Titration~~ Titrimetry
Fish sauce	**Sodium chloride and** Salt determined as chloride expressed as sodium chloride	~~20 (NaCl) Minimum limit~~ **From 20 (NaCl)** **From** 12 (Cl^-)	18 **- 22** 11 **- 13**	2.0 1.2	4.0 2.4	5.1 5.5	98–102 98–102	NMKL 178 AOAC 971.27 AOAC 976.18 AOAC 937.09	~~Potentiometric titration~~ Titrimetry (Potentiometric) ~~Potentiometric titration~~ Titrimetry (Potentiometric) ~~Titration~~ Titrimetry (Potentiometric) ~~Titration~~ Titrimetry
Salted Atlantic herring and salted sprat	**Sodium chloride and Salt determined as Chloride expressed as Sodium chloride**	**From 1 to 20 (NaCl)** **From 0.6 to 12 (Cl^-)**	**0.9 – 22** **0.5 - 13**	**0.1** **0.06**	**0.2** **0.12**	**8.0** **8.6**	**97-103**	**NMKL 178** **AOAC 971.27** **AOAC 976.18** **AOAC 937.09**	**Titrimetry (Potentiometric)** **Titrimetry (Potentiometric)** **Titrimetry (Potentiometric)** **Titrimetry**

Commodity	Provision	ML (%)	Min. appl. range (%)	LOD (%)	LOQ (%)	Precision (RSD_R) (%) no more than	Recovery (%)	Examples of applicable methods that meet the criteria	Principle
Salted Fish and dried salted fish of Gadidae family of fishes	Sodium chloride and Salt determined as Chloride expressed as Sodium chloride	From 12 (NaCl) From 7.3 (Cl^-)	11 – 13 6.8 – 8.1	1.2 0.8	2.4 1.5	5.5 5.9	98-102	NMKL 178 AOAC 971.27 AOAC 976.18 AOAC 937.09	Titrimetry (Potentiometric) Titrimetry (Potentiometric) Titrimetry (Potentiometric) Titrimetry
Sturgeon Caviar	Sodium chloride and Salt determined as Chloride expressed as Sodium chloride	From 3 to 5 (NaCl) From 1.8 to 3.0 (Cl^-)	2.7 -5.5 1.7 – 3.4	0.3 0.2	0.6 0.4	6.8 7.3	97-103	NMKL 178 AOAC 971.27 AOAC 976.18 AOAC 937.09	Titrimetry (Potentiometric) Titrimetry (Potentiometric) Titrimetry (Potentiometric) Titrimetry

1.3 FOODS FOR SPECIAL DIETARY USES

Foods for special dietary uses				
Commodity	**Provision**	**Method**	**Principle**	**Type**
Follow-up formula	**Vitamin A palmitate (retinyl palmitate), Vitamin A acetate (retinyl acetate)**	**AOAC 2012.10 / ISO 20633**	**HPLC-UV**	**II**
Follow-up formula	Vitamin A ~~(retinol isomers)~~	AOAC 992.04	HPLC**-UV**	~~II~~ **III**
Follow-up formula	Vitamin A ~~(retinol) (above 500 IU/l milk after reconstitution)~~	AOAC 992.06	HPLC**-UV**	III
~~Follow-up formula~~	~~Vitamin A~~	~~AOAC 974.29~~	~~Colorimetry~~	~~IV~~
Follow-up formula	**Vitamin E**	**AOAC 2012.10 / ISO 20633**	**HPLC-UV**	**II**
Follow-up formula	**Vitamin D**	**AOAC 2016.05 / ISO 20636**	**UHPLC-MS/MS**	**II**
Follow-up formula	**Thiamine**	**AOAC 2015.14 / ISO 21470**	**Enzymatic digestion and UHPLC-MS/MS**	**II**
Follow-up formula	**Riboflavin**	**AOAC 2015.14 / ISO 21470**	**Enzymatic digestion and UHPLC-MS/MS**	**II**
Follow-up formula	**Niacin**	**AOAC 2015.14 / ISO 21470**	**Enzymatic digestion and UHPLC-MS/MS**	**II**
Follow-up formula	**Vitamin B_6**	**AOAC 2015.14 / ISO 21470**	**Enzymatic digestion and UHPLC-MS/MS**	**II**
Follow-up formula	**Vitamin B_{12}**	**AOAC 2011.10 / ISO 20634**	**HPLC-VIS**	**II**
Follow-up formula	**Vitamin B_{12}**	**AOAC 2014.02**	**HPLC-UV**	**III**

Foods for special dietary uses				
Commodity	**Provision**	**Method**	**Principle**	**Type**
Follow-up formula	**Pantothenic acid**	**AOAC 2012.16 / ISO 20639**	**UHPLC-MS/MS**	**II**
Follow-up formula	Pantothenic acid	AOAC 992.07 ~~**Measures total pantothenate (free pantothenic acid + CoA- + ACP-bound) and measured as D-pantothenic acid (or calcium D-pantothenate)**~~	Microbioassay	~~**II**~~ **III**
Follow-up formula	**Folic Acid**	**AOAC 2011.06 / ISO 20631**	**HPLC-MS/MS**	**II**
Follow-up formula	**Vitamin C**	**AOAC 2012.22 / ISO 20635**	**UHPLC-UV**	**II**
Follow-up formula	**Biotin**	**AOAC 2016.02 / ISO 23305**	**HPLC-UV**	**II**
Follow-up formula	**Iron**	**AOAC 2015.06 / ISO 21424 | IDF 243**	**ICP-MS**	**II**
Follow-up formula	**Iron**	**AOAC 2011.14 / ISO 15151 | IDF 229**	**ICP-OES**	**III**
Follow-up formula	**Calcium**	**AOAC 2015.06 / ISO 21424 | IDF 243**	**ICP-MS**	**II**
Follow-up formula	**Calcium**	**AOAC 2011.14 / ISO 15151 | IDF 229**	**ICP-OES**	**III**
Follow-up formula	**Phosphorus**	**AOAC 2015.06 / ISO 21424 | IDF 243**	**ICP-MS**	**II**
Follow-up formula	**Phosphorus**	**AOAC 2011.14 / ISO 15151 | IDF 229**	**ICP-OES**	**III**
Follow-up formula	**Magnesium**	**AOAC 2015.06 / ISO 21424 | IDF 243**	**ICP-MS**	**II**

Foods for special dietary uses				
Commodity	**Provision**	**Method**	**Principle**	**Type**
Follow-up formula	Magnesium	AOAC 2011.14 / ISO 15151 | IDF 229	ICP-OES	III
Follow-up formula	Sodium	AOAC 2015.06 / ISO 21424 | IDF 243	ICP-MS	II
Follow-up formula	Sodium	AOAC 2011.14 / ISO 15151 | IDF 229	ICP-OES	III
Follow-up formula	Chloride	AOAC 2016.03 / ISO 21422 | IDF 242	Potentiometry	II
Follow-up formula	Potassium	AOAC 2015.06 / ISO 21424 | IDF 243	ICP-MS	II
Follow-up formula	Potassium	AOAC 2011.14 / ISO 15151 | IDF 229	ICP-OES	III
Follow-up formula	Manganese	AOAC 2015.06 / ISO 21424 | IDF 243	ICP-MS	II
Follow-up formula	Manganese	AOAC 2011.14 / ISO 15151 | IDF 229	ICP-OES	III
Follow-up formula	Iodine	AOAC 2012.15 / ISO 20647 | IDF 234	ICP-MS	II
~~Follow-up formula~~	~~Iodine (milk-based formula)~~	~~AOAC 992.24~~	~~Ion-selective potentiometry~~	~~II~~
Follow-up formula	Selenium	AOAC 2011.19 / ISO 20649 | IDF 235	ICP-MS	II
Follow-up formula	Copper	AOAC 2015.06 / ISO 21424 | IDF 243	ICP-MS	II
Follow-up formula	Copper	AOAC 2011.14 / ISO 15151 | IDF 229	ICP-OES	III
Follow-up formula	Zinc	AOAC 2015.06 / ISO 21424 | IDF 243	ICP-MS	II

Foods for special dietary uses				
Commodity	**Provision**	**Method**	**Principle**	**Type**
Follow-up formula	**Zinc**	**AOAC 2011.14 / ISO 15151 | IDF 229**	**ICP-OES**	**III**
Follow-up formula	**Total nucleotides**	**AOAC 2011.20 / ISO 20638**	**~~LC~~ SPE -HPLC-UV**	**II**
Follow-up formula	**Choline**	**AOAC 2015.10 / ISO 21468**	**UHPLC-MS/MS**	**II**
Follow-up formula	**Myo-inositol**	**AOAC 2011.18 / ISO 20637**	**HPLC-PAD**	**II**
Follow-up formula	**L-carnitine**	**AOAC 2015.10 / ISO 21468**	**UHPLC-MS/MS**	**II**
Follow-up formula	**Total amino acids (excluding taurine and tryptophan) for use according to section 3.1.3 (a) notes 2) and 3) of CXS 156-1987**	**AOAC 2018.06 / ISO 4214 | IDF 254 / AACC 07-50.01**	**UHPLC-UV**	**II**
Follow-up formula	**Tryptophan** **For use according to Section 3.1.3 (a) notes 2 and 3 of CXS 156-1987**	**AOAC 2017.03**	**HPLC-FLD**	**II**
Follow-up formula	**Total fatty acids**	**AOAC 2012.13 / ISO 16958 | IDF 231**	**GC-FID**	**II**
Follow-up formula	**Total fatty acids**	**AOAC 996.06**	**GC**	**III**
Follow-up formula	**Crude protein**	**ISO 8968-1 | IDF 20-1**	**Titrimetry (Kjeldahl digestion)**	**I**
Infant formula	Folic acid	AOAC 2011.06 / **ISO 20631**	**UHP**LC-MS/MS	II

Table 2. Methods of analysis for dietary fibre: *Guidelines for use of nutrition and health claims* (CXG 23-1997): Table of conditions for claims

Standard	Provisions	Method	Principle	Type
General methods that measure both the higher (monomeric units > 9) and the lower molecular weight fraction (monomeric units <=9) [(2)]				
All foods (1)	**Method applicable for determining the content of insoluble and soluble dietary fibres of higher and lower molecular weight. The method is applicable in food that may, or may not, contain resistant starches**	**AOAC 2022.01/ AACC 32-61.01/ ICC Standard No. 191****	**Enzymatic-gravimetry and HPLC**	**Type I**
~~**All foods (1)**~~	~~**Method applicable for determining the content of insoluble and soluble dietary fibres of higher and lower molecular weight. The method is applicable in food that may, or may not, contain resistant starches**~~	~~**AACC Intl 32-50.01 AOAC 2011.25**~~	~~**Enzymatic gravimetry High Pressure Liquid Chromatography**~~	~~**Type I**~~

**** Isolated, purified, and/or synthetic fibres captured by AOAC 2022.01/ICC Standard 191/AACC 32-61.01 that do not meet the Codex definition of dietary fibre in the *Guidelines on nutrition labelling* (CXG 2-1985) should be subtracted from the final measurement, where deemed appropriate by competent authorities.**

1.4 FRUIT JUICES AND NECTARS

Fruit juices and nectars				
Commodity	**Provision**	**Method**	**Principle**	**Type**
Fruit juices and nectars	Ascorbic acid-L ~~**(additives)**~~	IFU~~**MA**~~ 17~~**A**~~**a**	HPLC	II
Fruit juices and nectars	Ascorbic acid-L ~~**(additives)**~~	AOAC 967.21 **/** ~~**IFUMA 17**~~ ISO 6557-2	Indophenol method	III
Fruit juices and nectars	Ascorbic acid-L ~~**(additives)**~~	IFU~~**MA**~~ 17**b**	~~**Indophenol**~~ **Iodine** method	III
Fruit juices and nectars	Ascorbic acid-L ~~**(additives)**~~	ISO 6557-1	Fluorescence spectrometry	IV
~~**Fruit juices and nectars**~~	~~**Carbon dioxide (additives and processing aids)**~~	~~**IFUMA 42**~~	~~**Titrimetry (back-titration after precipitation)**~~	~~**IV**~~
Fruit juices and nectars	Citric acid[xviii] ~~**(additives)**~~	AOAC 986.13	HPLC	II
Fruit juices and nectars	**High Fructose Corn Syrup** ~~**HFCS**~~ and **Hydrolyzed Inulin Syrup** ~~**HIS**~~ in apple juice (permitted ingredients)	Determination of HFCS and HIS by Capillary GC method JAOAC 84~~,~~.486 (2001) **/ IFU recommendation No. 4**	~~**CAP**~~ GC ~~**method**~~	IV
~~**Fruit juices and nectars**~~	~~**Malic acid-L**~~	~~**EN 1138**~~	~~**Enzymatic determination**~~	~~**II**~~
Fruit juices and nectars	Malic acid-L	IFU~~**MA**~~ 21	Enzymatic determination	II
Fruit juices and nectars	Saccharin	NMKL 122	~~**Liquid chromatography**~~ **HPLC**	II
Fruit juices and nectars	Soluble solids	AOAC 983.17 **/** EN 12143 **/** IFU~~**MA**~~ 8 **/** ISO 2173	Indirect by refractometry	I

[xviii] All juices except citrus based juices.

Fruit juices and nectars				
Commodity	**Provision**	**Method**	**Principle**	**Type**
Fruit juices and nectars	Sucrose ~~**(permitted ingredients)**~~	EN 12146 **/** IFU~~**MA** 56~~	Enzymatic determination	III
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~ **Phosphorus / phosphate**	~~**Determination of phosphorus/phosphate**~~ EN 1136 **/** IFU~~**MA No** 50~~	Photometric determination	II

1.5 MILK AND MILK PRODUCTS

Milk and milk products				
Commodity	**Provision**	**Method**	**Principle**	**Type**
Butter	**Salt (Determined as chloride expressed as NaCl)**	**AOAC 2016.03 / ISO 21422 | IDF 242**	**Titrimetry (Potentiometric)**	**III**
Butter	Salt **(Determined as chloride expressed as NaCl)**	ISO 15648 | IDF 179	~~**Potentiometry (determination of chloride, expressed as sodium chloride)**~~ **Titrimetry (Potentiometric)**	II
Cheese	**Sodium Chloride (Determined as chloride, expressed as NaCl)**	**AOAC 2016.03 / ISO 21422 | IDF 242**	**Titrimetry (Potentiometric)**	**III**
Cheese	Sodium chloride **(Determined as chloride expressed as NaCl)**	ISO 5943 | IDF 88	~~**Potentiometry (determination of chloride, expressed as sodium chloride)**~~ **Titrimetry (Potentiometric)**	II
Whey powders	**Water[xlii] (moisture)[***]**	**Described in Appendix XI**	**Gravimetry (drying at 102°C)**	**IV**

[xlii] Water content excluding the crystallized water bound to lactose (generally known as moisture content).

[***] Due to accessibility to equipment and calibration of the method ISO 5537 | IDF 26, the method as described in Appendix XI is listed as Type IV. In a dispute situation, the Type I method shall be used. This 102ºC method is less precise, and results may not be consistent with results obtained with ISO 5537 | IDF 26, in particular for powders with high natural lactose such as whey powders.

APPENDIX XI

DETERMINATION OF MOISTURE IN POWDERED MILK, POWDERED CREAM, WHEY POWDER AND BLEND OF SKIMMED MILK POWDER WITH VEGETABLE FAT

TEST MOISTURE METHOD AT NORMAL PRESSURE (102 ± 2)°C IN POWDERED MILK, POWDERED CREAM, WHEY POWDER AND BLEND OF SKIMMED MILK POWDER WITH VEGETABLE FAT

DESCRIPTION OF THE METHOD: DETERMINATION OF MOISTURE

1. SCOPE

This ~~standard~~ **description** 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**, as well as whey powders**.

Table 3: Numeric performance criteria for methods of analysis for copper and iron in milk fat products

(Note: the numeric performance criteria are not for adoption or revocation. The only changes in underlined and/or ~~strikethrough~~ font are amendments / removal of example methods / principles)

Commodity	Provision	ML (mg/kg)	LOD (mg/kg)	LOQ (mg/kg)	RSDR (%)	Recovery	Minimum applicable range		Examples of applicable methods that meet the criteria	Principle
							Minimum	Maximum		
Milk fat products	Copper	0.05	0.010	0.020	44.0	60–115%	0.028	0.072	AOAC 2015.06 / ISO 21424 | IDF 243	ICP**-MS** ~~**mass spectrometry**~~
									~~**ISO 5738 | IDF 76**~~	~~**Photometry, (diethyldithiocarbamate)**~~
									~~**AOAC 960.40**~~	~~**Photometry, (diethyldithiocarbamate)**~~
Milk fat products	Iron	0.2	0.020	0.040	40.8	80–110%	0.08	0.32	AOAC 2015.06 / ISO 21424 | IDF 243	ICP**-MS** ~~**mass spectrometry**~~

1.6 MISCELLANEOUS PRODUCTS

Miscellaneous products				
Commodity	**Provision**	**Method**	**Principle**	**Type**
Dried meat	**Chloride as sodium chloride**	**AOAC 935.47 and AOAC 937.09B**	**Titrimetry (Volhard method)**	**III**
~~**Food grade**~~ **Food grade** salt	Iodine	WHO/UNICEF/ICCIDD method[xlv] Only applicable to a product which has been fortified with iodate	Titrimetry ~~using sodium thiosulphate~~	IV
Food grade salt	**Sodium chloride**	**See Appendix * Part A**	**Calculation**	**I**

PART B – METHODS OF SAMPLING BY COMMODITY CATEGORIES AND NAMES

Commodity categories	Method of sampling	Notes
Miscellaneous products		
Food grade salt	**See Appendix * Part B**	

[xlv] Assessment of iodine deficiency disorders and monitoring their elimination. A guide for programme managers. Third edition, Annex 1: Titration method for determining salt iodate and salt iodine content. World Health Organization, Geneva, 2007. The report is available from ~~http://www.who.int/nutrition/publications/micronutrients/iodine_deficiency/WHO_NHD_01.1/en/index.html~~ **https://www.who.int/publications/i/item/9789241595827**

APPENDIX *

DETERMINATION OF SODIUM CHLORIDE AND RELATED SAMPLING METHOD FOR FOOD GRADE SALT

Part A. DETERMINATION OF SODIUM CHLORIDE CONTENT

This method allows the calculation of sodium chloride content, as provided for in CXS 150 Section 3.1, on the basis of the results of the determinations of sulphate, calcium and magnesium, potassium and loss on drying. Convert sulphate to $CaSO_4$ and unused calcium to $CaCl_2$, unless sulphate in sample exceeds the amount necessary to combine with calcium, in which case convert calcium to $CaSO_4$ and unused sulphate first to $MgSO_4$ and any remaining sulphate to Na_2SO_4. Convert unused magnesium to $MgCl_2$. Convert potassium to KCl. Convert unused halogens to NaCl. Report the NaCl content on a dry matter basis, multiplying the percentage NaCl by 100/100-P, where P is the percentage loss on drying.

Part B. SAMPLING: METHOD FOR THE SAMPLING OF FOOD GRADE SALT FOR THE DETERMINATION OF SODIUM CHLORIDE

1. SCOPE

This method specifies the sampling procedure to be applied when determining the main component in order to assess the food grade quality of sodium chloride (salt) as provided for in the Codex Standard for Food Grade Salt, Section 3: "Essential Composition and Quality Factors".

The criterion to be used for acceptance or rejection of a lot or consignment on the basis of this sample is also provided.

2. FIELD OF APPLICATION

This method is applicable to the sampling of any type of salt intended for use as food, either prepacked or in bulk.

3. PRINCIPLE

This method represents a variables sampling procedure for mean quality: blended bulk sample analysis.

A blended bulk sample is produced in such a way that it is representative of the lot or consignment. It is composed of a proportion of items drawn from the lot or consignment to be analyzed.

Acceptance criterion is on the basis that the mean value obtained from analyses of those blended bulk samples must comply with the provision in the Standard.

4. DEFINITIONS

The terms used in this sampling method refer to those in the "*General Guidelines on Sampling"* (CXG 50-2004) unless stated otherwise.

5. EQUIPMENT

The sampling equipment used should be adapted to the nature of the tests to be carried out (for example: sampling by borer, sampling equipment made of chemically inert material, etc.). The containers used for collecting the samples should be made of a chemically inert material and should be air-tight.

6. PROCEDURE

6.1 PREPACKED SALT

Sampling may be carried out by "random sampling" or by "systematic sampling". The choice of the method to be used depends on the nature of the lot (for example: if the packages are marked with successive numbers, systematic sampling may be suitable).

6.1.1 Random sampling

Draw the n items from the lot in such a way that each item in the lot has the same chance of being selected.

6.1.2 Systematic sampling

If the N units in the lot have been classified and can be numbered from 1 to N, the 1-in-k systematic sampling of n items can be obtained as follows:

a) Determine the k value as k = N/n. (If k is not an integer, then round to the nearest integer).

b) From the first k items in the lot take one at random and then take every k^{th} item thereafter.

6.2 SALT IN BULK

Here, the lot is fictitiously divided into items (strata); a lot with a total mass of m kg is considered to be composed of m/100 items. In this case, it is necessary to draw up a "stratified sampling" plan appropriate to the lot dimension. The samples are selected from all the strata in proportion to the stratum sizes.

Note: Stratified sampling of a population which can be divided into different subpopulations (called strata) is carried out in such a way that specified proportions of the sample are drawn from the different strata.

6.3 CONSTITUTION OF THE SAMPLE

6.3.1 The size and the number of the items forming the sample depend on the type of salt and the lot magnitude. The minimum size to be taken into account should be in accordance with one of the following specifications according to the circumstances:

- 250 g of salt in bulk or prepacked in more than 1 kg packages;
- one package for prepacked salt in 500 g or 1 kg packages.

The appropriate number of samples to be drawn from the lot, shall be determined in accordance with "*General Guidelines on Sampling*" (CXG 50-2004).

6.3.2 Combine and mix well the different items drawn from the lot. This blended bulk sample constitutes the laboratory sample. More than one laboratory sample may be composed in such a manner.

7. ACCEPTANCE CRITERION

7.1 Determine the NaCl content (%) of at least two test portions of the laboratory sample.

7.2 Calculate the average of the results obtained for the n test portions of the laboratory sample using:

$$\bar{x} = \frac{\sum x}{n} (n \geq 2)$$

7.3 In accordance with the provision for the relevant NaCl content (%), a lot or a consignment shall be considered acceptable if the following condition is verified:

$\bar{x} \geq$ minimum level specified.

8. SAMPLING REPORT

The sampling report should contain the following information:

a) type and origin of the salt;
b) alterations of state of the salt (e.g. presence of foreign matter);
c) date of sampling;
d) lot or consignment number;
e) method of packing;
f) total mass of lot or consignment
g) number, unit mass of packages and whether the mass is given net or gross;
h) number of items sampled;
i) number, nature and initial position of sampled items;
j) number, composition and mass of the bulk sample(s) and the method used to obtain and conserve it (them);
k) names and signatures of the people who carried out the sampling.

Part 2

METHODS OF ANALYSIS WHICH REMAIN UNCHANGED IN CXS 234 AS A RESULT OF DECISIONS BY CCMAS44

2.1 CEREALS, PULSES AND LEGUMES AND DERIVED PRODUCTS

Cereals, pulses and legumes and derived products				
Commodity	**Provision**	**Method**	**Principle**	**Type**
Quinoa	Protein	ISO 1871	Titrimetry (Kjeldahl digestion)	IV

2.2 FRUIT JUICES AND NECTARS

Fruit juices and nectars				
Commodity	**Provision**	**Method**	**Principle**	**Type**
Fruit juices and nectars	Malic acid (additives)	AOAC 993.05	Enzymatic determination and HPLC	III
Fruit juices and nectars	Preservatives in fruit juices (sorbic acid and its salts)	ISO 5519	Spectrometry	III

2.3 MILK AND MILK PRODUCTS

Milk and milk products				
Commodity	**Provision**	**Method**	**Principle**	**Type**
Whey powders	Water[xlii] (moisture)	ISO 5537 | IDF 26	Gravimetry (drying at 87°C)	I

2.4 MISCELLANEOUS PRODUCTS

Miscellaneous products				
Commodity	**Provision**	**Method**	**Principle**	**Type**
Dried meat	Chloride as sodium chloride (≥ 1.0%)	ISO 1841-1	Titrimetry (Volhard method)	III
Dried meat	Chloride as sodium chloride (≥ 0.25%)	ISO 1841-2	Titrimetry (potentiometry)	II

[xlii] Water content excluding the crystallized water bound to lactose (generally known as moisture content).

Part 3

METHODS OF ANALYSIS FOR FURTHER CONSIDERATION

3.1 COCOA PRODUCTS AND CHOCOLATE (For further consideration by the EWG on cocoa products and chocolate)

Note: Text indicated in ~~strikethrough~~, or **bold** and underlined font indicate changes and/or additions discussed in relation to the method of analysis as it currently appears in CXS 234-1999.

Cocoa products and chocolate				
Commodity	**Provision**	**Method**	**Principle**	**Type**
Chocolate and chocolate products	Cocoa butter **(determined as fat)**	**ICA No. 26 / AOAC 977.10 and** AOAC 963.15 **/ ICA ~~IOCCC~~** 14	**Calculation from moisture (Determined as Water) and** Gravimetry (Soxhlet extraction)	I
Chocolate and chocolate products	Milk fat	**~~IOCCC~~ ICA No.** 5 **~~AOAC 945.34; 925.41B; 920.80~~**	Titrimetry/Distillation	**~~I~~ IV**
Chocolate and chocolate products	Moisture	**~~IOCCC 26 or AOAC 977.10 (Karl Fischer method); or~~** AOAC 931.04 or **~~IOCCC~~ ICA No.** 1	Gravimetry **- drying at 100-102°C**	**~~I~~ IV**
Chocolate and chocolate products	**Moisture (Determined as Water)**	**ICA No. 26 / AOAC 977.10**	**Titrimetry - Karl Fischer**	**II**
Chocolate and chocolate products	Non-cocoa butter vegetable fat	AOCS Ce 10/02 **~~and described in the standard~~**	**~~Described in the standard~~ GC-MS**	**~~I~~ IV**
Cocoa (cacao) mass or cocoa/ chocolate liquor, and cocoa cake	Fat	**ICA No. 26 / AOAC 977.10 and** AOAC 963.15 **/ ~~or~~ ~~IOCCC~~ ICA No.** 14	**Calculation from moisture (Determined as Water) and** Gravimetry (Soxhlet extraction)	I
Cocoa butter	Free fatty acids	ISO 660 **~~or~~ /** AOCS Cd 3d-63	Titrimetry	I
Cocoa butter	Unsaponifiable matter	ISO 3596 **~~or~~ /** ISO 18609 **~~or~~ /** AOCS Ca 6b-53	Titrimetry after extraction with diethyl ether	I
Cocoa powders (cocoa) and dry cocoa-sugar mixtures	Moisture **(Determined as Water)**	**~~IOCCC~~ ICA No.** 26 **~~or~~ /** AOAC 977.10 **~~(Karl Fischer method)~~**	~~Gravimetry~~ **Titrimetry - Karl Fischer**	~~I~~ **II**

Cocoa products and chocolate				
Commodity	**Provision**	**Method**	**Principle**	**Type**
Chocolate and chocolate products	**Cocoa butter equivalents in cocoa butter and plain chocolate**	**ISO 23275-1 and ISO 23275-2 / AOCS Ce 11-05**	**GC-FID**	**I**
Chocolate and chocolate products	**Cocoa Butter Equivalents in Milk Chocolate**	**ISO 11053 / AOCS Ce 11a-07**	**GC-FID**	**I**
Chocolate and chocolate products	**Determination of centre and coating of filled chocolate**	**See Appendix ****		
Cocoa powders (cocoas) and dry mixtures of cocoa and sugars	**Determination of full-fat cocoa powder, fat-reduced cocoa powder and highly fat-reduced cocoa powder**	**AOAC 977.04 or IOCCC 26 (1988)-Karl Fisher Method**		
Cocoa powders (cocoas) and dry mixtures of cocoa and sugars	**Determination of cocoa butter**	**To be developed**		

APPENDIX **: DETERMINATION OF CENTRE AND COATING OF FILLED CHOCOLATE IN CHOCOLATE AND CHOCOLATE PRODUCTS

All methods approved for the chocolate type used for the coating and those approved for the type of centre concerned.

3.2 FOODS FOR SPECIAL DIETARY USES (For CCNFSDU's consideration)

Foods for special dietary uses				
Commodity	**Provision**	**Method**	**Principle**	**Type**
Follow-up formula	Vitamin A	EN 12823-1 (all-trans-retinol and 13-cis-retinol) Vitamin A (both natural + supplemental ester forms) aggregated and quantified as individual retinol isomers (13-cis and all-trans)	HPLC-UV or FL	III
Follow-up formula	Vitamin E	EN 12822 (Measures vitamin E (both natural + supplemental ester forms) aggregated and quantified as individual tocopherol congeners (α, β, γ, δ)	HPLC-UV or FL	III
Follow-up formula	Vitamin E	AOAC 992.03 Measures all rac-vitamin E (both natural + supplemental ester forms) aggregated and quantified as α-congeners	HPLC-UV	III
Follow-up formula	Vitamin D	EN 12821 / NMKL 167 (D2 and/or D3 measured as single components. Hydroxylated forms not measured)	HPLC-UV	III
Follow-up formula	Vitamin D	AOAC 995.05 D2 and D3 measured	HPLC-UV	III
Follow-up formula	Thiamine	AOAC 986.27****	Fluorimetry	III
Follow-up formula	Thiamine	EN 14122 (Measures all vitamin B1 forms (natural and added free, bound and phosphorylated) following extraction and conversion to thiamine)	HPLC-FL (with pre-or post-column derivatization to thiochrome)	III
Follow-up formula	Riboflavin	EN 14152 (Measures natural and supplemental forms, free, bound and phosphorylated (FMN and FAD) aggregated and measured as riboflavin)	HPLC-FL	III

**** Care should be taken in the application of the method due to spectral interference.

Foods for special dietary uses				
Commodity	**Provision**	**Method**	**Principle**	**Type**
Follow-up formula	Riboflavin	AOAC 985.31[****]	Fluorimetry	III
Follow-up formula	Niacin	EN 15652 (Free and bound and phosphorylated forms measured either as aggregate of nicotinic acid + nicotinamide, or as individual forms)	HPLC-FL (with post-column photochemical derivatization)	III
Follow-up formula	Niacin	AOAC 985.34 (niacin (preformed) and nicotinamide)	Microbioassay and turbidimetry	III
Follow-up formula	Vitamin B_6	EN 14166 (Aggregates free and bound pyridoxal, pyridoxine and pyridoxamine and measures as pyridoxine)	Microbioassay	III
Follow-up formula	Vitamin B_6	AOAC 985.32	Microbioassay	III
Follow-up formula	Vitamin B_6	AOAC 2004.07 / EN 14164 (Free and bound phosphorylated forms (pyridoxal, pyridoxine and pyridoxamine) converted and measured as pyridoxine)	HPLC-FL	III
Follow-up formula	Vitamin B_{12}	AOAC 986.23 (Measures total vitamin B12 as cyanocobalamin)	Turbidimetry	III
Follow-up formula	Folic acid	EN 14131 (Total folate (free + bound), aggregated and measured as folic acid)	Microbioassay	III
Follow-up formula	Folic acid	AOAC 992.05 (Measures free folic acid + free, unbound natural folates, aggregated, and measured as folic acid)	Microbioassay	III
Follow-up formula	Biotin	EN 15607 (d-biotin) (Measures total D-biotin [free + D-biocytin])	HPLC- FLD (post-column derivatization)	III

[****] Care should be taken in the application of the method due to spectral interference.

Foods for special dietary uses				
Commodity	**Provision**	**Method**	**Principle**	**Type**
Follow-up formula	Iron	AOAC 985.35	FAAS	III
Follow-up formula	Iron	AOAC 999.11|NMKL 139	FAAS	III
Follow-up formula	Calcium	ISO 8070|IDF 119	FAAS	III
Follow-up formula	Calcium	AOAC 985.35	FAAS	III
Follow-up formula	Phosphorus	AOAC 986.24	Spectrophotometry	III
Follow-up formula	Magnesium	ISO 8070|IDF 119	FAAS	III
Follow-up formula	Magnesium	AOAC 985.35	FAAS	III
Follow-up formula	Sodium	ISO 8070|IDF 119	FAAS	III
Follow-up formula	Chloride	AOAC 986.26	Potentiometry	III
Follow-up formula	Potassium	ISO 8070|IDF 119	FAAS	III
Follow-up formula	Manganese	AOAC 985.35	FAAS	III
Follow-up formula	Selenium	AOAC 2006.03	ICP-OES	III
Follow-up formula	Selenium	EN 14627	HGAAS	III
Follow-up formula	Selenium	AOAC 996.16	Fluorimetry	III
Follow-up formula	Selenium	AOAC 996.17	HGAAS	III

Foods for special dietary uses				
Commodity	**Provision**	**Method**	**Principle**	**Type**
Follow-up formula	Copper	AOAC 985.35	FAAS	III
Follow-up formula	Zinc	AOAC 985.35	FAAS	III
Follow-up formula	Choline	AOAC 999.14	Enzymatic colorimetric method with limitations on applicability due to choline and ascorbate concentration	III

3.3 FRUIT JUICES AND NECTARS (For further consideration by the Expert Group)

Note: Text indicated in ~~strikethrough~~, or **bold** and underlined font indicate changes and/or additions discussed in relation to the method of analysis as it currently appears in CXS 234-1999.

Fruit juices and nectars				
Commodity	**Provision**	**Method**	**Principle**	**Type**
Fruit juices and nectars	Ascorbic acid-L **~~(additives)~~**	ISO 6557-2**: 1995**	**~~Indophenol method~~** **A) Titrimetry** B) **(for strongly coloured) Spectrometry**	**~~III~~** **IV**
Fruit juices and nectars	Citric acid[xviii] **~~(additives)~~**	AOAC 986.13 **(1996)**	HPLC	II
~~Fruit~~ Apple juice**~~s and nectars~~**	Malic acid-D in apple juice	AOAC 995.06 **(1998)**	HPLC	II
Fruit juices and nectars	Quinic, malic and citric acid in cranberry juice cocktail and apple juice (permitted ingredients and additives)	**~~Determination of quinic, malic and citric acid in cranberry juice cocktail and apple juice~~** AOAC 986.13 **(1986)**	HPLC	III
Fruit juices and nectars	Sucrose **~~(permitted ingredients)~~**	EN 12630 IFU**~~MA~~** 67 **(2005) /** NMKL 148 **(1993)**	HPLC	II
Fruit juices and nectars	Cellobiose	IFU**~~MA~~ Recommendation No.** 4 **October 2000**	Capillary **~~gas chromatography~~ GC**	IV
Fruit juices and nectars	Citric acid[xxix] **~~(additives)~~**	EN 1137 IFU**~~MA~~** 22 **(2005)**	Enzymatic determination	III
Fruit juices and nectars	Glucose-D and fructose-D **~~(permitted ingredients)~~**	**~~EN 1140~~** IFU**~~MA~~** 55 **(2005)**	Enzymatic determination	II
Fruit juices and nectars	Malic acid-D	**~~EN 12138~~** IFU**~~MA~~** 64 **(2005)**	Enzymatic determination	II
Fruit juices and nectars	Pectin **~~(additives)~~**	IFU**~~MA~~** 26 **(2012)**	Precipitation/photometry	I

[xviii] All juices except citrus based juices.
[xxix] All juices except citrus based juices.

Fruit juices and nectars				
Commodity	**Provision**	**Method**	**Principle**	**Type**
Fruit juices and nectars	Benzoic acid and its salts; sorbic acid and its salts	IFU~~MA~~ 63 **(2005)** NMKL 124 **(1997)**	HPLC	II
Fruit juices and nectars	Benzoic acid and its salts	ISO 5518**:2011**, ISO 6560**:1983**	Spectrometry	III
Fruit juices and nectars	Sulphur dioxide ~~**(additives)**~~	Optimized Monier-Williams AOAC 990.28 **(2005)** IFU~~MA~~ 7A **(2018)** NMKL 132 **(1989)**	Titrimetry after distillation	II
Fruit juices and nectars	Sulphur dioxide ~~**(additives)**~~	NMKL 135 **(1990)**	Enzymatic determination	III
Fruit juices and nectars	Sulphur dioxide ~~**(additives)**~~	ISO 5522**:1995**~~,~~ ISO 5523**:1995**	Titrimetry after distillation	III
Fruit juices and nectars	Tartaric acid in grape juice ~~**(additives)**~~	~~**EN 12137**~~ IFU~~MA~~ 65 **(2005)**	HPLC	II
Fruit juices and nectars	Total nitrogen	~~**EN 12135**~~ IFU~~MA~~ 28 **(2005)**	Digestion/titration	I
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Acetic acid (acetate)**	~~**Determination of acetic acid**~~ ~~**EN 12632;**~~ IFU~~MA~~ 66 **(2019)**	Enzymatic determination	II
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Alcohol (ethanol)**	~~**Determination of alcohol (ethanol)**~~ IFU~~MA~~ 52 **(2005)**	Enzymatic determination	II

[xx] **3.4 Verification of composition, quality and authenticity**

Fruit juices and nectars should be subject to testing for authenticity, composition and quality where applicable and where required. The analytical methods used should be those found in Section 9 (Methods of analysis and sampling).

The verification of a sample's authenticity/quality can be assessed by comparison of data for the sample, generated using appropriate methods included in the standard, with that produced for fruit of the same type and from the same region, allowing for natural variations, seasonal changes and for variations occurring due to processing.

Fruit juices and nectars				
Commodity	**Provision**	**Method**	**Principle**	**Type**
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Anthocyanins**	~~**Detection of anthocyanins**~~ IFU~~**MA**~~ 71 **(2023)**	HPLC	I
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Ash**	~~**Determination of ash in fruit products**~~ AOAC 940.26 **(1940): 525°C;** ~~**EN 1135;**~~ IFU~~**MA**~~ 9 **(2005): 500-550°C**	Gravimetry	I
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Beet sugar**	~~**Detection of beet sugar in fruit juices**~~ AOAC 995.17 **(1998)**	~~**Deuterium**~~ **SNIF-**NMR	II
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Benzoic acid**	~~**Determination of benzoic acid as a marker in orange juice**~~ AOAC 994.11 **(1964)**	HPLC	III
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **C^{13}/C^{12} ratio of ethanol derived from fruit juices**	~~**Determination of C^{13}/C^{12} ratio of ethanol derived from fruit juices**~~ JAOAC 79, No. 1, 1996, 62-72	Stable isotope mass spectrometry	II
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Carbon stable isotope ratio**	~~**Determination of carbon stable isotope ratio of apple juice**~~ AOAC 981.09 **(1997)**~~**–**~~ JAOAC 64, 85 (1981)	Stable isotope mass spectrometry	II
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Carbon stable isotope ratio**	~~**Determination of carbon stable isotope ratio of orange juice**~~ AOAC 982.21 **(1997)**	Stable isotope mass spectrometry	II
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Carotenoid, total/individual groups**	~~**Determination of carotenoid, total/individual groups**~~ ~~**EN 12136;**~~ IFU~~**MA**~~ 59 **(2008)**	Spectrophotometry	I
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Centrifugable pulp**	~~**Determination of centrifugable pulp**~~ ~~**EN 12134;**~~ IFU~~**MA**~~ 60 **(2005)**	Centrifugation/% value	I

Fruit juices and nectars				
Commodity	**Provision**	**Method**	**Principle**	**Type**
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Chloride (expressed as sodium chloride)**	~~**Determination of chloride (expressed as sodium chloride)**~~ ~~**EN 12133**~~ IFU~~**MA**~~ 37 **(2005)**	Electrochemical titrimetry	III
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Chloride**	~~**Determination of chloride in vegetable juice**~~ AOAC 971.27 **(1996)** ~~**(Codex general method)**~~ ISO 3634**:1995**	Titration	II
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Essential oils**	~~**Determination of essential oils (Scott titration)**~~ AOAC 968.20 **(1969)** – IFU~~**MA**~~ 45 **(2005)**[xxi]	(Scott) distillation, titration	I
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Essential oils in citrus fruit**	~~**Determination of essential oils (in citrus fruit) (volume determination)**~~エラー**!** ブックマークが定義されていません。 ISO 1955**:1995**	Distillation and direct reading of the volume determination	I
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Fermentability**	~~**Determination of fermentability**~~ IFU~~**MA**~~ 18 **(1998)**	Microbiological method	I
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Formol number**	~~**Determination of formol number**~~ ~~**EN 1133**~~ IFU~~**MA**~~ 30 **(2005)**	Potentiometric titration	I
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Free amino acids**	~~**Determination of free amino acids**~~ ~~**EN 12742**~~ IFU~~**MA**~~ 57 **(2005)**	~~**Liquid chromatography**~~ **LC**	II
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Fumaric acid**	~~**Determination of fumaric acid**~~ IFU~~**MA**~~ 72 **(1998)**	HPLC	II

[xxi] Because there is no numerical value in the standard, duplicate Type I methods have been included which may lead to different results.

Fruit juices and nectars				
Commodity	**Provision**	**Method**	**Principle**	**Type**
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Glucose, fructose, saccharose**	~~**Determination of glucose fructose and saccharose**~~ ~~**EN 12630**~~ IFU~~**MA**~~ 67 **(2005)** NMKL 148 **(1993)**	HPLC	II or III
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Gluconic acid**	~~**Determination of gluconic acid**~~ IFU~~**MA**~~ 76 **(2006)**	Enzymatic determination	II
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Glycerol**	~~**Determination of glycerol**~~ IFU~~**MA**~~ 77 **(2005)**	Enzymatic determination	II
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Hesperidin and naringin**	~~**Determination of hesperidin and naringin**~~ ~~**EN 12148**~~ IFU~~**MA**~~ 58 **(2005)**	HPLC	II
~~**Fruit**~~ **Apple** juice~~**s and nectars**~~	**High Fructose Corn Syrup and Hydrolyzed Inulin Syrup** ~~**HFCS and HIS in apple juice (permitted ingredients)**~~	~~**Determination of HFCS and HIS by Capillary GC method**~~ JAOAC 84, 486 (2001)	~~**CAP**~~ GC ~~**method**~~	IV
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Hydroxymethylfurfural**	~~**Determination of hydroxymethylfurfural**~~ IFU~~**MA**~~ 69 **(2005)**	HPLC	II
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Hydroxymethylfurfural**	~~**Determination of hydroxymethylfurfural**~~ ISO 7466**:1986**	Spectrometry	III
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Isocitric acid-D**	~~**Determination of isocitric acid-D**~~ IFU~~**MA**~~ 54 **(2005)**	Enzymatic determination	II
Fruit juices and nectars	Isocitric acid-D	EN 1139 (1999)	Enzymatic determination	II

Fruit juices and nectars				
Commodity	**Provision**	**Method**	**Principle**	**Type**
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Lactic acid- D and L**	~~**Determination of Lactic acid- D and L EN 12631**~~ IFU~~**MA**~~ 53 **(2005)**	Enzymatic determination	II
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **L-malic/total malic acid ratio – to detected added D-malic acid**	~~**Determination of L-malic/total malic acid ratio in apple juice**~~ AOAC 993.05 **(1997)**	Enzymatic determination and HPLC	II
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Naringin and Neohespiridin**	~~**Determination of naringin and neohesperidin in orange juice**~~ AOAC 999.05 **(2002)**	HPLC	III
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **pH - value**	~~**Determination of pH value**~~ NMKL 179 **(2005)**	Potentiometry	II
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **pH - value**	~~**EN 1132**~~ IFU~~**MA**~~ 11 **(2015)** ISO 1842**:1995**	Potentiometry	IV
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Proline**	~~**Determination of proline by photometry – non-specific determination EN 1141**~~ IFU~~**MA**~~ 49 **(2005)**	Photometry	I
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Relative density**	~~**Determination of relative density EN 1131 (1993);**~~ IFU~~**MA 0**~~1 **(2005)** & IFU Method No General sheet (1971)	Pycnometry	II
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Relative density**	~~**Determination of relative density**~~ IFU~~**MA**~~ 01A **(2005)**	Densitometry	III
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Sodium, potassium, calcium, magnesium**	~~**Determination of sodium, potassium, calcium, magnesium in fruit juices EN 1134**~~ IFU~~**MA**~~ 33 **(2005)**	~~**Atomic absorption spectroscopy**~~ **AAS**	II

Fruit juices and nectars				
Commodity	**Provision**	**Method**	**Principle**	**Type**
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Sorbitol-D**	~~**Determination of sorbitol-D**~~ IFU~~**MA**~~ 62 **(2005)**	Enzymatic determination	II
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Stable carbon isotope ratio**	~~**Determination of stable carbon isotope ratio in the pulp of fruit juices ENV 13070**~~ Analytica Chimica Acta 340 (1997)	Stable isotope mass spectrometry	II
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Stable carbon isotope ratio of sugars from fruit juices**	~~**Determination of stable carbon isotope ratio of sugars from fruit juices ENV 12140**~~ Analytica Chimica Acta 271 (1993)	Stable isotope mass spectrometry	II
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx]	~~**Determination of stable hydrogen isotope ratio of water from fruit juices ENV 12142**~~	~~**Stable isotope mass spectrometry**~~	~~**II**~~
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx]	~~**Determination of stable oxygen isotope ratio in fruit juice water ENV 12141**~~	~~**Stable isotope mass spectrometry**~~	~~**II**~~
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Starch**	~~**Detection of starch**~~ AOAC 925.38 **(1925)** IFU~~**MA**~~ 73 **(2000)**	Colorimetric	I
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Sugar beet derived syrups in frozen concentrated orange juice**	~~**Determination of sugar beet derived syrups in frozen concentrated orange juice $\delta^{18}O$ Measurements in water**~~ AOAC 992.09 **(1997)**	Oxygen isotope ratio analysis **($\delta^{18}O$ in water)**	I
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Titratable acids**	~~**Determination of titrable acids, total EN 12147**~~ IFU~~**MA**~~ 03 **(2017)** ISO 750**:1998**	Titrimetry	I

Fruit juices and nectars				
Commodity	**Provision**	**Method**	**Principle**	**Type**
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Total dry matter at 70°C**	~~**Determination of total dry matter (vacuum oven drying at 70 °C)**~~[xxii] ~~**EN 12145**~~ IFU~~**MA** 61~~ **(2005)**	Gravimetry~~**ic determination**	I
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Total solids (Microwave oven drying)**	~~**Determination of total solids (microwave oven drying)**~~エラー! ブックマークが定義されていません。 AOAC 985.26 **(2001)**	Gravimetry~~**ic determination**	I
Fruit juices and nectars	~~**Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**~~[xx] **Vitamin C (dehydro-ascorbic acid and ascorbic acid)**	~~**Determination of vitamin C (dehydro-ascorbic acid and ascorbic acid)**~~ AOAC 967.22 **(1968)**	Microfluorometry	III

[xxii] Because there is no numerical value in the standard, duplicate Type I methods have been included which may lead to different results.

APPENDIX III

NITROGEN TO PROTEIN CONVERSION FACTORS FOR COMMODITIES APPROVED BY COMMODITY COMMITTEES

(For approval by CAC48 for inclusion as an Annex in CXS 234-1999)

Animal Protein Source

Milk and milk products - 6.38

Meat and meat products - 6.25

Infant formula - The calculation of the protein content of infant formulas prepared ready for consumption should be based on N x 6.25, unless a scientific justification is provided for the use of a different conversion factor for a particular product. The value of 6.38 is generally established as a specific factor appropriate for conversion of nitrogen to protein in other milk products, and the value of 5.71 as a specific factor for conversion of nitrogen to protein in other soy products.

Follow-up formula for older infants and product for young children - The calculation of the protein content of the final product ready for consumption should be based on N x 6.25, unless a scientific justification is provided for the use of a different conversion factor for a particular product. The protein levels set in this standard are based on a nitrogen conversion factor of 6.25. For information the value of 6.38 is used as a specific factor appropriate for conversion of nitrogen to protein in other Codex standards for milk products.

In accordance with the *Guidelines on nutrition labelling* (CXG 2-1985), the calculation of protein for nutrient declaration purposes should be based on a conversion factor of 6.25, unless a different factor is specified in the present annex.

Fish and fishery products

Crackers from marine and freshwater fish, crustaceans and molluscan shellfish - 6.25

Plant Protein Source

Wheat, wheat protein products - 5.71

Maize - 6.25

Quinoa - 6.25

Sorghum - 6.25

Millet (grains and flour) - 5.71

Gochujang - 6.25

Soya and non-fermented soybean products - 5.71

Tempe - 5.71

Natto - 5.71

Cheonggukjang - 5.71

Thua Nao – 5.71

Vegetable protein Products (VPP): Products produced by separation from wheat and soya grains and flours of certain non-protein constituents (starch, other carbohydrates) - 6.25

Soy protein products - 6.25

Appendix IV

Information document for the *General guidelines on sampling* (CXG 50-2004)

(For publication on the Codex webpage)

Contents

1 Introduction

The purpose of this document is to provide additional information on the sampling plans referred to in CXG 50, including background and examples for each of the main types of sampling plan. A link to the app1 for the design and evaluation of these sampling plans is included. Links for new apps will be included when they become available.

It should be noted that CXG 50 does not request to use a specific sampling plan. For import/export quarantine, it is important for the government to choose an appropriate sampling plan for commodity/provision combinations taking into account the feasibility, scientific information, and administrative necessity.

The document consists of three parts:

Part 1 consists of Section 2 and Section 3 and contains general information relating to the design of sampling plans, including examples:

Section 2.1 deals with principles behind the “classical” approach to sampling plans based on specification of producer’s and consumer’s risks, to allow for any level nonconforming in a lot.

Section 2.2 contains information about the design process, including suggestions on the use of pre-defined sampling plans, such as ISO plans, as well as specifications of allowable risks, as a starting point.

Section 2.3 describes the different apps that were provided with the original package and provides a link to an on-line app for the design of attribute and variables sampling plans with and without measurement uncertainty.

Section 3 contains case studies showing the main types of sampling plans mentioned in CXG 50, including some where measurement uncertainty is non-negligible.

Sections 3.1 and 3.2 deal with various types of attributes and variables plans, including an explanation of the basis underlying the plans in the ISO2859 and ISO3951 standards.

Section 3.3 discusses sampling for bulk materials with a particular focus on the plans for mycotoxins described in the *General standard for contaminants and toxins in food and feed* (CXS 193-1995).

Section 3.4 covers other sampling plans. Examples include attribute sampling plans with an AQL of 6.5 taken from ISO, and ad hoc plans using small numbers of samples formed into composite samples for testing.

Part 2 consists of Section 4 and Section 5 and contains more background on sampling plans including a statistical appendix:

Section 4 includes statistical derivation of attributes and variables plans when measurement uncertainty is negligible and references the sampling plans in Part 1.

Section 4.3 discusses measurement uncertainty and its role in acceptance sampling.

Section 4.6.5 covers the basis for sampling plans for Mycotoxins derived by Whitaker et al. that are special cases of plans for bulk materials and outlines the statistical complexity for the future development of sampling plans for bulk materials.

Section 4.7 contains information about other sampling plans, including 3-class attribute plans, used for microbiological assessments.

Part 3 consists of Section 6 and provides information regarding Bayesian sampling plans.

Section 6.2 discusses Bayesian plans based on a risk-based approach. More specifically, the plans are based on the concepts of specific consumer risk and conformance probability from JCGM 106. An overview of Bayesian risks is also provided. This approach was developed in ISO TC 69 SC 5 WG 10 and is described in a technical report as well as in a separate publication.

Section 6.3 discusses Bayesian plans based on a utility-based approach. Standard plans are provided for the practitioner. This approach was developed in ISO TC 69 SC 5 WG 10 and is described in a technical report as well as in a separate publication.

Note:

Some Excel formulas in the text (and in the Excel file provided) use the English style, with decimal points and comma separators.

PART ONE

General information relating to the design of sampling plans

Examples and case studies

2 Design of sampling plans

2.1 Principles behind the design of sampling plans

2.1.1 Producers and consumers

Depending on the nature of the transaction, a producer could include either:

- A manufacturer, supplier or seller of a food product or ingredients, or
- A regulatory agency providing assurance for exported product to an importing country agency.

and a consumer could include:

- A customer purchasing the food product or ingredient for the manufacture of other food products, or
- An importing country regulatory agency seeking to provide assurance to the individual consumers living in that country, or
- An exporting country regulatory agency providing official assurance to an importing country agency acting on behalf of the importing country, or
- An individual purchasing a food product, although individuals would not normally be able to carry out lot-based inspections of foods, or
- A manufacturer purchasing ingredients for the production of a food product.

2.1.2 Producer's and consumer's risks

Acceptance sampling plans always carry intrinsic risks that a lot of poor quality will be incorrectly accepted or that a lot of good quality will be incorrectly rejected. These two risks are generally referred to as the consumer's risk and the producer's risk, respectively.

However, by following statistical principles sampling plans can be designed to control these risks to allowable levels. This is achieved by specifying a particular producer's risk quality level, the PRQ, and a particular consumer's risk quality level, the CRQ, along with a corresponding producer's risk (PR), the probability of rejecting a lot with quality level equal to the PRQ, and a consumer's risk (CR), the probability of accepting a lot with quality level is equal to the CRQ, respectively. Once these four parameters, the (PRQ, CRQ, PR and CR), are specified the sampling plan is uniquely determined and the probability of acceptance and therefore the producer's and consumer's risks at any quality level can be calculated.

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

The *Principles for the use of sampling and testing in international food trade* (CXG 83-2013) recommends that ideally, producers and consumers should agree on a sampling plan prior to its use. However, direct collaboration/negotiation between producers and consumers on the sampling plan to be used or the way in which it will be used might not always be possible.

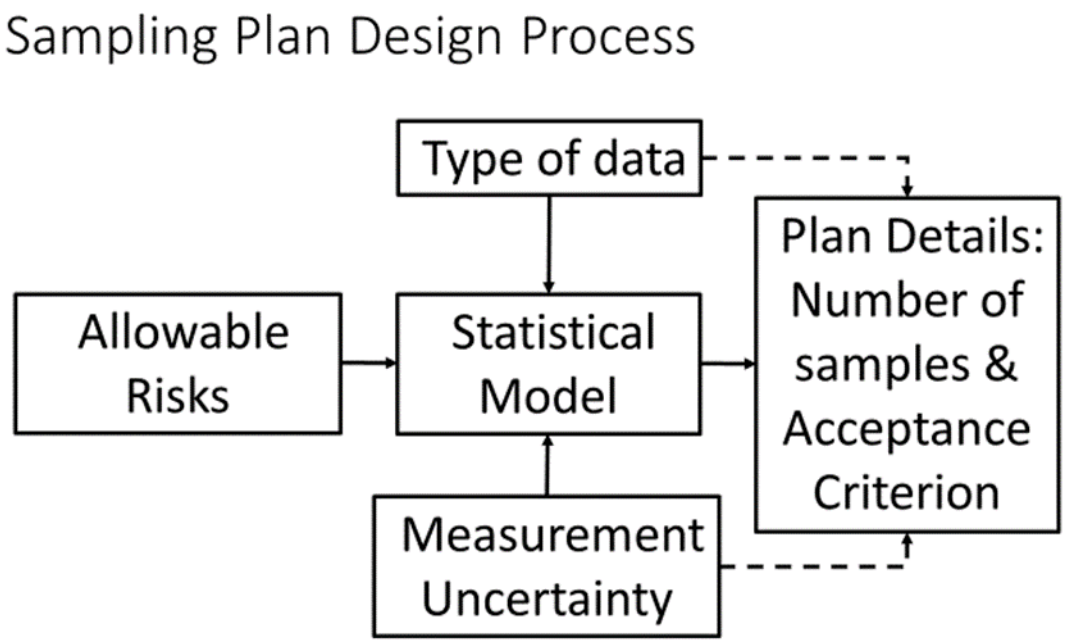
This is the traditional approach to the design of sampling plans. In this approach, risks are calculated on the basis of a known quality level, e.g. "if a lot contained $X\%$ of nonconforming items, then the probability of acceptance would be P."

However, it is also possible to introduce a Bayesian framework which allows other definitions of risk, involving the use of a prior distribution based e.g. on past inspections of a characteristic in a product. Bayesian methods can potentially allow the user to achieve a considerable reduction in sample size.

2.2 Design of sampling plans

2.2.1 Overview of the design process

Figure 1 Sampling Plan Design Process



This diagram shows a high-level view of the design process, showing the fundamental inputs to the design as reflected in the workflow in the *General guidelines on sampling* (CXG 50-2004).

Specification of the allowable risks is a key input, the producer's risk and the consumer's risk might both be specified or only one of those risks might be specified. In the ISO plans only the consumer's risk would be specified for the inspection of isolated lots of incoming goods whereas plans based on only the producer's risk might be used in the context of a long-term supply contact between a manufacturer and a customer.

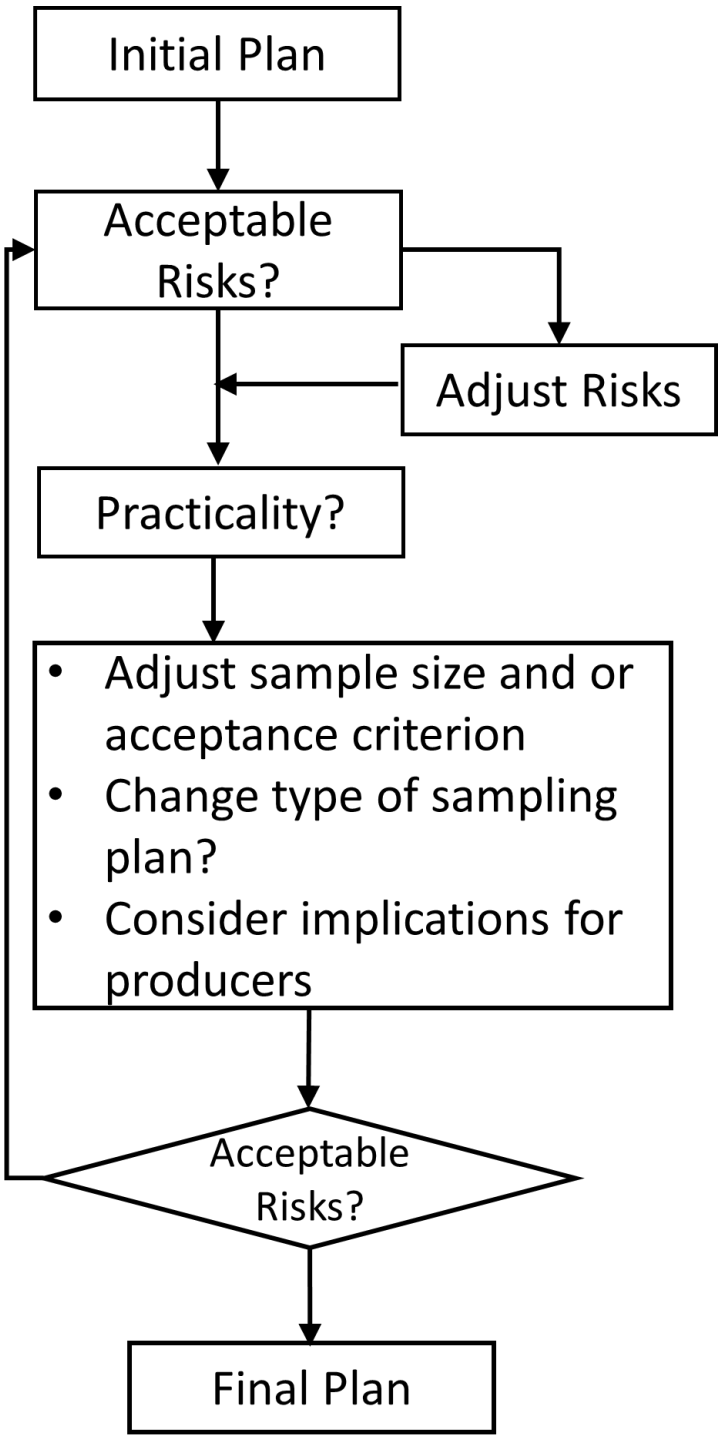
The type of data and non-negligible measurement uncertainty will determine the statistical model that is used to work out the details of the sampling plan. For example, if one has attributes data the statistical model is based on the binomial distribution, whereas if the one has variables data the model could be based on the normal distribution or in the case of a compositional proportion the beta distribution, or possibly some other distribution (not dealt with in CXG 50-2004).

The type of data and the presence of non-negligible measurement uncertainty also determine the form of the acceptance criterion; in the simplest case where measurement uncertainty is negligible an attributes plans is specified by the number of samples n and the acceptance number c, but a variables plan is specified by the number of samples n and the acceptability constant k. The form of the acceptance criterion could be more complex for variables plans where measurement uncertainty is non-negligible.

2.2.2 Process for the design of sampling plans

This diagram shows a process that might be used to design a sampling plan.

Figure 2 Process for Design of Sampling Plans



Step 1 Select an Initial Plan as a starting point:

The design of sampling plans requires specification of the allowable consumer's and producer's risks following which sampling plans are designed using statistical methodology.

However, it is often difficult for designers of plans to decide on appropriate levels of allowable risks [that should possibly be decided jointly by both the producer and the consumer], so that the following process is suggested as a way of proceeding.

The starting point could be a plan from either ISO 2859-1 or ISO 3951-1, depending on whether one has attributes or variables data. In CXG 50-2004 Appendix 1 plans with selected PRQ and CRQ levels were used as starting points.

The following plans based on these ISO standards could be used as starting points for the design of plans. In these plans the producer's risk (PR) is 5% and the consumer's risk (CR) is 10%.

Table: Attributes plans from ISO 2859-1 for PRQ = 6.5%

PRQ	c	n	CRQ
6.5%	0	2	68.4%
6.5%	0	3	53.6%
6.5%	1	5	58.4%
6.5%	1	8	40.6%
6.5%	2	13	36.0%
6.5%	3	20	30.4%
6.5%	5	32	27.1%
6.5%	7	50	22.4%

Table: Attributes plans from ISO 2859-1 for PRQ = 1.5%

PRQ	c	n	CRQ
1.5%	0	8	25.0%
1.5%	0	13	16.2%
1.5%	1	20	18.1%
1.5%	1	32	11.6%
1.5%	2	50	10.3%

Table: Variables plans from ISO 3951-1 for PRQ = 2.5% (σ-method)

PRQ	k	n	CRQ
2.5%	1.115	3	35.4%
2.5%	1.240	6	23.7%
2.5%	1.419	8	16.7%
2.5%	1.366	8	18.1%
2.5%	1.370	12	15.9%
2.5%	1.439	16	13.2%
2.5%	1.456	21	12.0%
2.5%	1.533	29	9.76%
2.5%	1.606	42	7.95%

Step 2 Examine the OC curve:

A plan taken directly from a standard might not necessarily be suitable for a particular application, it might be too stringent or not stringent enough. Users need to consider whether the acceptance probability, the proportion of lots that will be accepted in the longer term by the plan, is acceptable at various levels nonconforming that might occur, for example:

- Is the probability of acceptance of lots containing 10% (or 5% or 20%) of nonconforming product acceptable?

Step 3 Adjust the Risks to the desired levels.

Step 4 Consider the practicality of the proposed sampling plan:

A key consideration is the number of samples that will need to be taken and tested for each lot that is inspected, and the expected number of lots that will be inspected in any year.

In general sample numbers can be economized by:

- Increasing the CRQ or decreasing the PRQ, or both
- Increasing the producer's and/or the consumer's risks PR and CR (both might be increased)
- Use of indifference quality plans for commodity characteristics (refer CXG 50-2004 3.2.2)
- Requiring a lower stringency at the individual lot level in favour of assurance in the longer term.

Specific ways to economize sample numbers include:

- Not assessing compliance of the lot on an individual basis but treating the product in the lot as a bulk material and inspecting the lot as a whole rather than focusing on compliance of the individual items
- Use of variables plans instead of attributes plans where applicable.

- Use of known lot standard deviations, if they are known.
- Use of plans based on the beta distribution for compositional characteristics.
- Use of offsets (including offsets to allow for non-negligible laboratory bias) (refer CXG 50-2004 3.2.3)
- Bayesian plans may be another way sample numbers might be reduced.

Step 5 Examine the OC curve to check that the risks are acceptable.

Step 6 Adopt the sampling plan or return to step 3 and repeat the process.

Measurement uncertainty should also be allowed for if it is non-negligible.

The resulting plan should be reviewed to ensure it will meet users' expectations and, where appropriate to ensure that it is fair to producers – choice of a suitable sampling plan should focus on the control of risks and the total cost, especially the costs of incorrect acceptance and incorrect rejection of lots, rather than just the cost of sampling and testing.

However, if multiple characteristics are inspected when assessing compliance to a standard, there is a risk that the producer's risk of inappropriate rejection will increase with the number of characteristics inspected. This risk can be mitigated by reducing the producer's risk on each of the individual sampling plans, so that the overall producer's risk is not excessive. This measure should be applied only among 'similar' characteristics, such as compositional characteristics.

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, remembering that in Codex, CXG 50-2004 will be applied mainly to compositional characteristics and commodity defects (refer CXG 50-2004 3.2.2 Fairness).

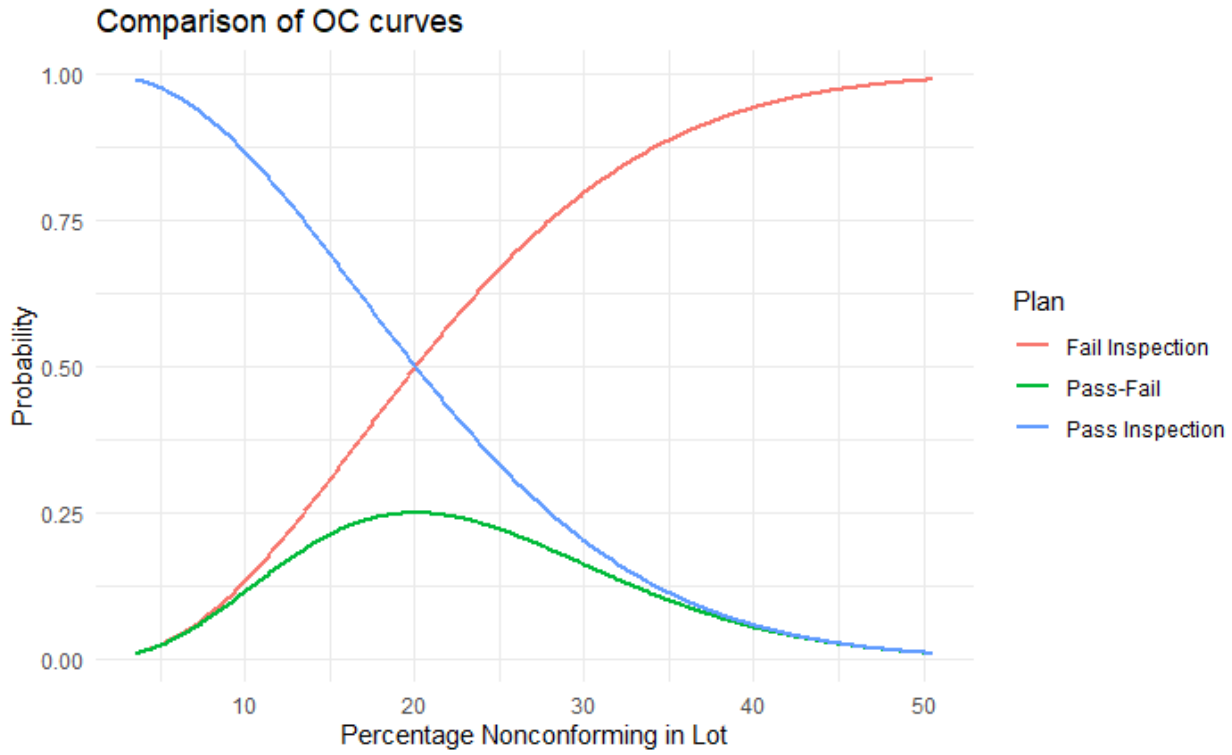
2.2.3 Use of the same sampling plan by the producer and the consumer

It is not always appropriate for a producer to use the same sampling plan as that used by a consumer, particularly if the consumer is also going to inspect the lot.

The following plot shows the probability of acceptance of a lot for a given plan [in **blue**] for the inspection and the probability of rejection using the same plan [in **red**]. The **green** line shows the probability that a lot will be accepted in the producer's inspection but then be rejected in the consumer's inspection. The maximum probability of a lot being accepted on an initial inspection and then being rejected on a subsequent inspection using the same plan is 25% that occurs when the probability of acceptance by the plan is 50%.

This shows that producers are potentially disadvantaged in adversarial sampling situations, where both the producer and the consumer both test the product; that should be considered in the design of plans to ensure fairness.

Figure 3 OC curves In Adversarial Sampling



The OC curves on the plot have been calculated as follows.

- The probability of acceptance (the blue line) is the usual calculation, described in section 3.1 for attributes plans and section 3.2 for variables plans for cases when the measurement uncertainty is negligible. If measurement uncertainty is non-negligible it is necessary to use the app.
- The probability of rejection (red line) is easily calculated since if a lot is either accepted or rejected:

$$Probability\ of\ rejection\ =\ 1\ -\ Probability\ of\ acceptance$$

- The probability of acceptance of a lot and subsequent rejection upon reinspection (the green line) is the product of the two probabilities:

$$Probability\ (Pass-Fail)\ =\ Probability\ of\ acceptance\ \times\ Probability\ of\ rejection$$

Obvious mitigations include:

- the producer using a plan that reduces the risk of rejection if the lot is inspected by a consumer,
- for the producer to operate at a quality level that ensures a lower rate of rejection if the lot is inspected by the consumer,
- consumers might rely on the producer's inspections rather than inspect the lots themselves.

2.2.4 CCMAS endorsement of sampling plans

Commodity Committees might propose sampling plans for provisions, or they might propose outcomes for sampling plans in terms of the maximum allowable producer's and consumer's risks for the inspection of a provision. This means that there is often more than one option for the sampling plan that could be used. The latter approach, specifying the outcome, is needed when measurement uncertainty is non-negligible as the plan will depend on the lot standard deviation, that will vary among producers.

The OIML International Recommendation R087[1] for the average quantity system in prepackaged is an example where maximum allowable producer's and consumer's risks are specified:

Producer's Risk

The probability of rejecting a lot whose true mean weight is equal to or exceeds the label quantity e.g. weight should be at most 0.5%.

[1] OIML Recommendation (OIML R 87): Quantity of product in prepackages, International Organisation for Legal Metrology, Paris (2016)

Consumer's Risk

The probability of accepting a lot whose true mean weight is less than the label quantity e.g. weight by more than a specified amount (not provided here) should be at most 10%.

These risk specifications are used in two ways to design plans for the inspection of quantities by weights, in a variables-plan to check compliance of the average weight and in an attributes plan to check that there is not an excessive proportion of deficient packages, weighing less than the label weight by more than a certain amount, in the lot.

2.3 Apps for the design and evaluation of sampling plans

This section contains a brief description of each of the three apps provided with the CXG 50-2004 package, along with links from where they can be run. References to the relevant sections in CXG 50-2004 are provided and further information can be found in this document.

The apps all follow the same general format and have been designed to:

- *Plan Evaluation* of a specified sampling plan to calculate the probability of acceptance in terms of the percentage nonconforming[2] in a lot and to show the Operating Characteristic (OC) curve, and to calculate the producer's and consumer's quality levels PRQ and CRQ corresponding to specified producer's and consumer's risks (with default values of 5% and 10% respectively)
- *Plan Design*, the design of a sampling plan, working out the number of samples and the acceptance criterion, the acceptance number 'c' or the acceptability constant 'k', from specifications of the producer's and consumer's risk quality levels and their associated probabilities of rejection and acceptance, respectively.

2.3.1 Description of apps

App1 relates to the design and evaluation of attributes sampling plans and variables plans for normally distributed characteristics, including situations where measurement uncertainty is also normally distributed. The app can be used to examine and compare the producer's and consumer's risks and the OC curves for different sampling plan options.

In attributes plans, the app can evaluate a sampling plan specified by a sample size 'n' and an acceptance number 'c' or design a plan based on the specified values of the PRQ, CRQ, producer's risk, and consumer's risk. The OC curves and the producer's and consumer's risks are shown for both plans.

Variables sampling plans are similar except there is a *k*-constant instead of an acceptance number. There is also an additional parameter, which is whether the standard deviation is known or unknown. The app also takes account of measurement uncertainty with the values of the components of measurement uncertainty and the lot standard deviation where these values have been specified.

This app has been deployed on the shinyapps.io server:

https://codex-testing.shinyapps.io/codex-testing-SamplingPlan/

Links for following apps will be included when they become available.

App2 relates to Fractional Nonconformance Plans and allows users to evaluate a specified plan, specified by the number of samples and the maximum allowable value of the individual sample FNC values for acceptance of the lot, or to design a sampling plan from specifications of consumer's and producer's risks.

See section 3.2.4 for further details.

App3 relates to plans for the assessment of lots for compositional characteristics. These plans are based on the beta distribution so that it is possible to apply the plan based on a single test of a composite sample formed from a specified number of increments. The app can evaluated a specified plan or design a plan based on specified risks. Refer to section 3.3.2 for details.

3 Case studies (examples for specific scenarios)

These examples follow the step-by-step design process described in CXG 50-2004 Appendix 1 and illustrate the use of the apps.

[2] The proportion of nonconforming items expressed as a percentage. Also called the proportion nonconforming in the ISO 2859 and 3951 standards.

3.1 Examples on the Use of Attributes plans

3.1.1 Example: Attributes plan with $c > 0$

In general, if both the consumer's and producer's risks are specified in the design of the plan, as might be appropriate for non-food safety characteristics such as commodity defects, it is unlikely that the acceptance numbers, the c values, will be zero. It should be noted that rather large sample sizes (and large acceptance numbers) might be needed for plans where the operating ratio (CRQ/PRQ) is small.

Example: Browning in Milk powder

- A customer found higher than usual levels of browning (discoloration) in a lot of WMP. The customer advised that they could accept the powder provided no more than 20% of the powder was nonconforming, as it would still be usable.
- The manufacturer wanted to control the risk of rejecting product that might still be usable, so that the product should be accepted [most of the time] if there was 10% nonconforming in the lot.

Key steps in the step-by-step design process

1. Attributes or variables data?

Excessive browning is an example of attributes data, samples are classified as either PASS or FAIL when compared against a reference powder.

2. Inspection error negligible or non-negligible?

It is assumed that inspection error is negligible in this example.

3. Set consumer's risk quality level (CRQ):

The customer has advised that the powder is still usable even if the level non-conforming was 20%, so that, for the purposes of the inspection, the consumer's risk quality level could be set at 20%.

4. Set producer's risk quality level (PRQ):

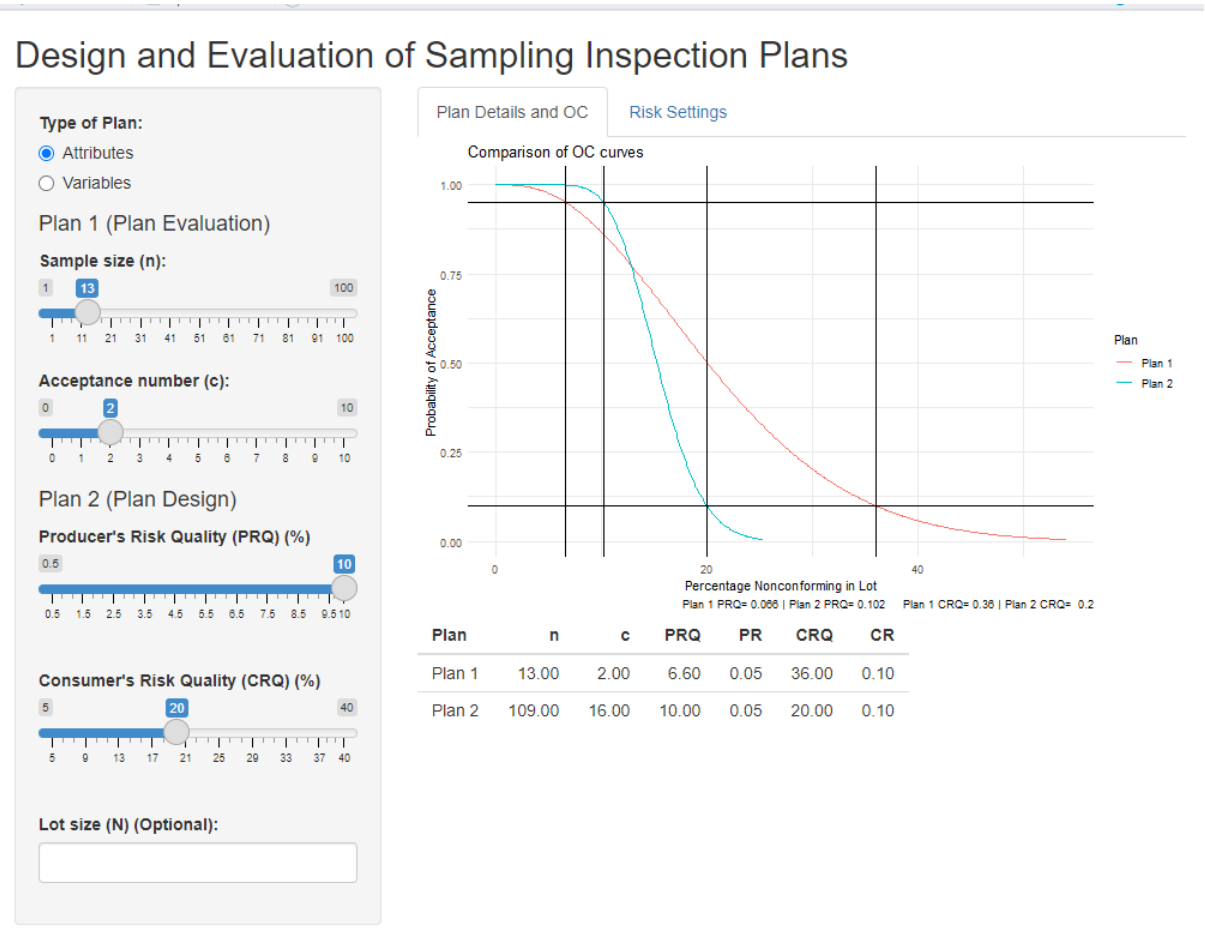
The PRQ can be set anywhere below the CRQ, noting that the smaller the operating ratio $\frac{CRQ}{PRQ}$, the larger the number of samples required to be taken.

Various values of the PRQ can be tried to assess the required sample size. Some possible options are:

PRQ	CRQ	n	c
5%	20%	38	4
10%	20%	109	16
15%	20%	500	88

For the purposes of this example, a producer's risk quality level of 10% was used; a PRQ of 5% is too stringent considering that the powder is still usable if up to 20% of the lot was nonconforming, and 500 samples is too many to take and evaluate.

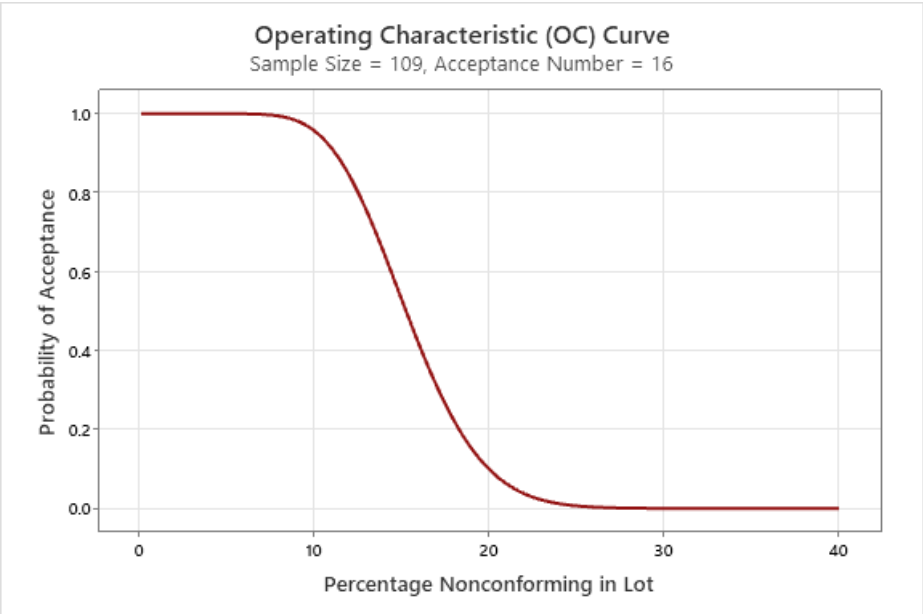
Figure 4 Design of plan for dispute resolution



Plan	n	c	PRQ	PR	CRQ	CR
Plan 1	13.00	2.00	6.60	0.05	36.00	0.10
Plan 2	109.00	16.00	10.00	0.05	20.00	0.10

The required sampling plan, required to control the consumer's and producer's risk to the specified levels is (n = 109, c=16) i.e. n=109 samples are taken, and the lot is accepted provided no more than 16 of those samples is nonconforming.

Figure 5 OC curve of selected plan - dispute resolution



The Operating Characteristic shows a 95% chance of accepting the lot when the level nonconforming is 10% (i.e. the PRQ), a 50% chance approximately of accepting the lot when the level nonconforming is 15% and a 10% chance of accepting the lot when the level nonconforming is 20% (the CRQ).

3.1.2 Example: Attributes plan with c=0

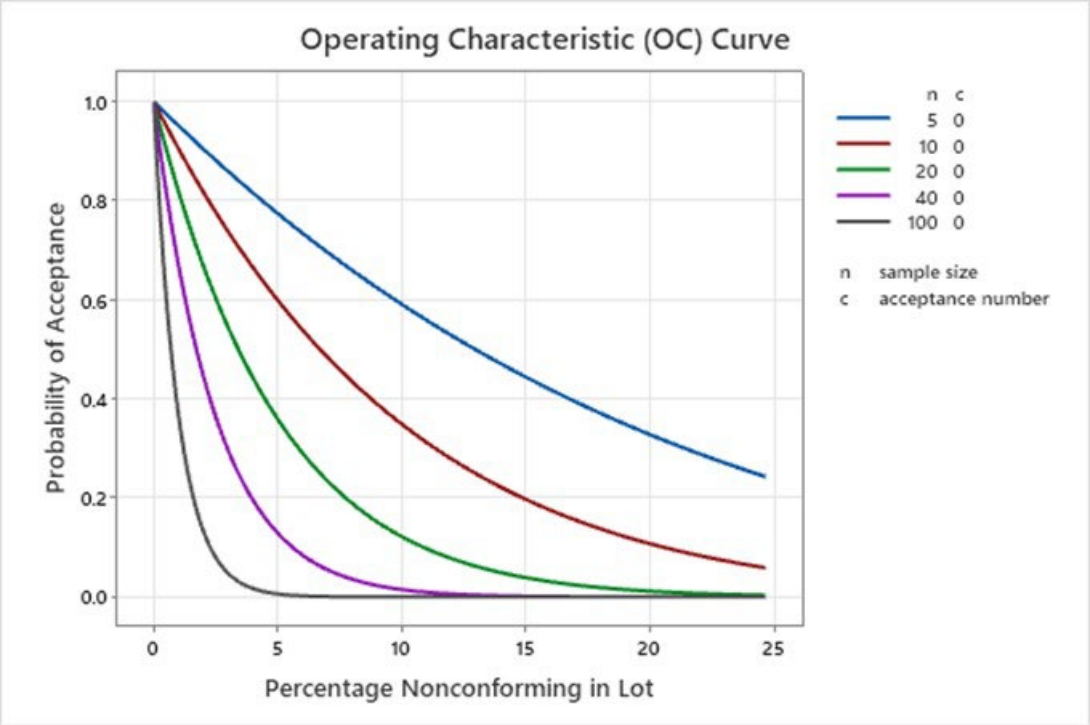
Refer to CXG 50-2004 4.2.5 Zero-acceptance number plans.

Zero-acceptance number (ZAN) plans are a special case of two-class plans in which the acceptance numbers are set to c = 0. These plans 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.

ZAN plans are commonly used, apparently based on the philosophy of zero defects and the perception that if c>0 then lots containing nonconforming product are being accepted.

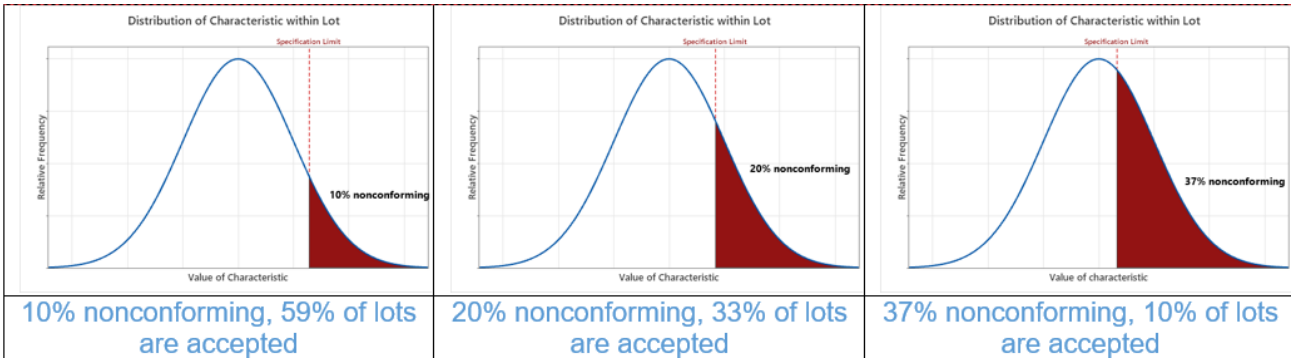
The following shows the Operating Characteristics for several ZAN plans:

Figure 6 OC curves for ZAN plans



However, ZAN plans cannot guarantee that lots that have passed inspection do not contain nonconforming items; no matter which plan is used there will always be a risk of accepting a lot containing some level of nonconforming product. The following shows the chance of accepting a lot for various levels nonconforming, using the (n=5, c=0) sampling plan.

Figure 7 Risks with n=5, c=0 plans



The number of samples n can be calculated directly using the formula:

$$CR = (1 - CRQ)^n \text{ or } n = \log(CR) / \log(1 - CRQ)$$

Typical outcomes that are often expressed in terms of the quality in the lot are as follows:

- If we select 60 items, taken at random from a 'lot', and find none of those items nonconforming, then we can claim with 95% confidence that no more than 5% of ALL the items in the lot are nonconforming.

- If we select 150 items, taken at random from a 'lot', and find none of those items nonconforming, then we can claim with 95% confidence that no more than 2% of ALL the items in the lot are nonconforming.
- If we select 300 items, taken at random from a 'lot', and find none of those items nonconforming, then we can claim with 95% confidence that no more than 1% of ALL the items in the lot are nonconforming.

If one or more nonconforming items has been found, it is still possible to make a statement about the quality level within the lot.

The Excel file pexact.xlsx included in the package can be used to calculate the 95% confidence intervals for the level nonconforming in a lot, or the total number of defects in a lot, for any number of nonconforming items or defects found in the sample, noting that an individual item may have more than a single defect:

- The sheet Binomial calculates 95% confidence intervals for the level of individual items conforming in the lot overall.
- The sheet Poisson calculates 95% confidence intervals for the number of defects in the lot overall. These limits can be converted to rates by dividing by the number of items examined.

Excel formulas for the calculation of the lower and upper 95% confidence limits for the two cases are given in Section 4.1.1.

Examples:

Binomial Case

If n=60 items were examined and c=2 of those 60 items were found nonconforming, then the estimated percentage of nonconforming items in the lot is 2/60 = 3.33% and, with 95% confidence, the level nonconforming in the lot lies between 0.41% and 11.53%.

Poisson Case

If 60 items were examined and 5 defects were found, with possibly more than one defect found on a single item, then with 95% confidence the number of defects in the lot lies between 1.62 and 11.67. Equivalently, these numbers could be expressed as defect rates of 1.62*100/60 = 2.7 to 11.67*100/60=19.45 defects per 100 items.

Example: Inspection for Foreign Matter

It is suspected that a lot is contaminated with foreign matter but that the contamination is not believed to be a food safety concern. However, it is known that the intended customer will not accept product in which any foreign matter has been found, so that a zero-acceptance number (ZAN) plan should be used.

Since the contamination is not a food-safety issue, it was decided to design a plan based on a consumer's risk (CR) of 5%, at a consumer's risk quality level (CRQ) of 3%.

The number of samples *n* can be calculated directly using the formula given above:

$CR = (1 - CRQ)^n$ or $n = \log(CR) / \log(1 - CRQ)$

Using the second formula we have:

$$n = \frac{\log(CR)}{\log(1 - CRQ)} = \frac{\log(0.05)}{\log(0.97)} = \frac{-2.9957}{-0.0305} = 98$$

Therefore, the lot is accepted provided none of the 98 samples inspected contains any foreign matter contamination. In practice one might use n=100 for simplicity.

3.1.2.1 (n=1, c=0) sampling plans

These sampling plans, often used by classifying variables data as attributes, are commonly used for inspection of contaminants and more widely, with or without allowance for MU.

For contaminants these plans rely on an assumption of homogeneity and possibly also on the usually large offsets between the decision limits used in those plans and food safety levels, so that allowance for MU may not be necessary. However, there is a considerable risk of incorrectly accepting a noncompliant lot if that lot was not homogeneous.

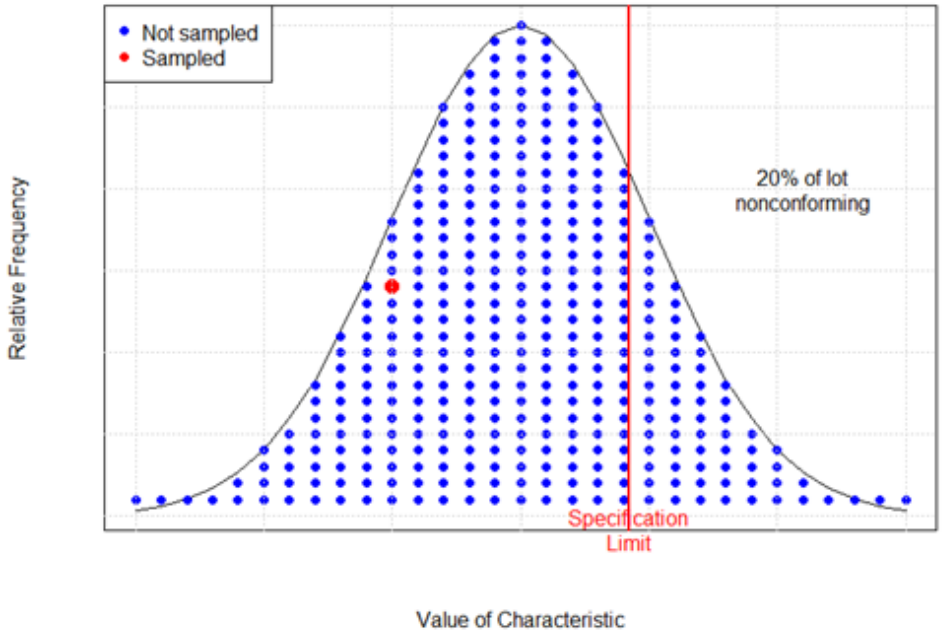
For other characteristics (n=1, c=0) plans are often used but might not provide the desired level of assurance to consumers. This might be due to:

- Attempting to minimize the cost of testing
- Ignoring of the principles of sampling

- Performing a spot check on compliance of the lot, that is potentially unfair to producers, without any intention of protecting consumer's risk; this practice often leads to complaints, there being a wide perception that if nonconforming items/samples are found on inspection then the entire lot is nonconforming

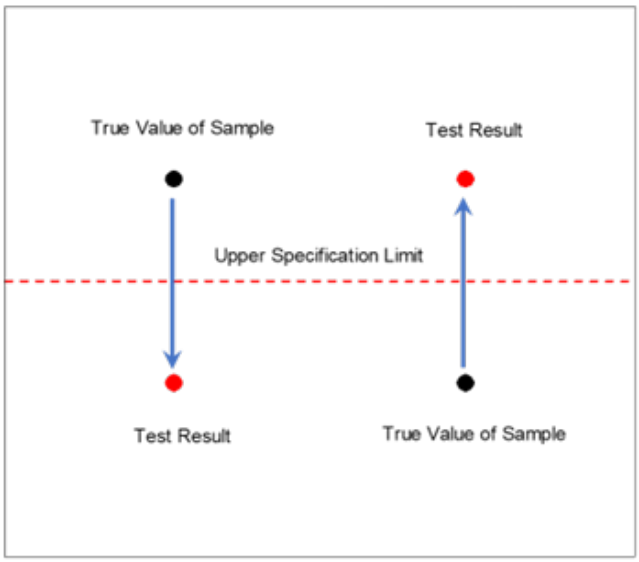
The fundamental problem with (n=1, c=0) plans is that decisions on acceptance or rejection are not necessarily related to the quality of the lots (Refer CXG 50-2004 Section 2.2). The following diagrams show the potential risks when using (n=1, c=0) sampling plans. The first plot shows the risk of making an incorrect decision due to sampling error, when 20% of the lot is nonconforming there is an 80% chance of not finding a nonconforming sample if only one sample is taken, assuming measurement uncertainty is negligible.

Figure 8 Risk due to sampling uncertainty for n=1 plans



The second plot shows the risk of making an incorrect decision due to analytical measurement uncertainty.

Figure 9 Risk due to measurement uncertainty in n=1 plans



The (n=1, c=0) plans have been extended to include allowance for MU [See CXG 54-2004 Figure 1 where MU uncertainty intervals are included to show the decision process.].

These plans, however, cannot simply be extended to lot inspection by including sampling components in the MU while allowing the PR and CR to be controlled to specified levels. Also, given that a single result is an estimate of the mean of a lot, use of this adjustment amounts to assessing compliance of the mean level of

the lot by comparing it against a maximum or minimum limit for the entire distribution. This comparison remains inappropriate regardless of whether sampling uncertainty is allowed for or not.

Sometimes guard-bands are applied, but their use may be unfair to producers.

If the characteristic is a compositional proportion, then provided measurement uncertainty is negligible, it is possible to design a sampling plan that can control both producer's and consumer's risks, but which requires only a single test of a composite sample to be performed.

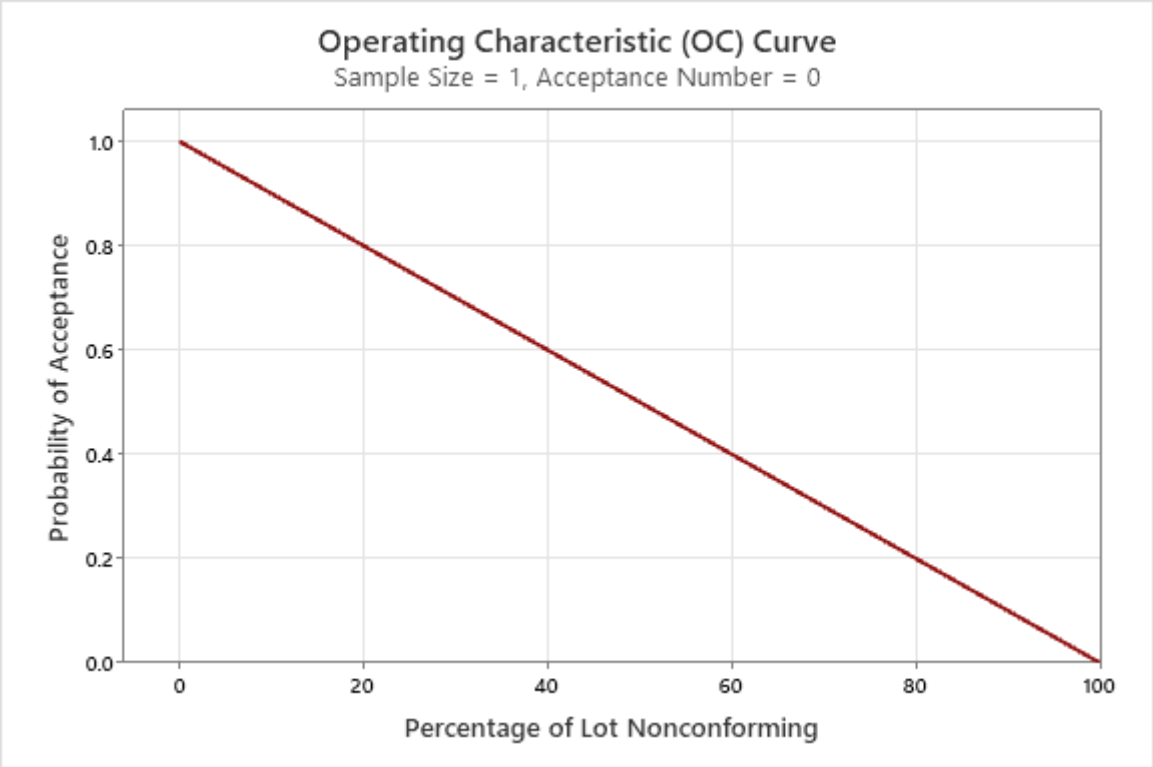
Refer to the example in section 3.3.2.

Section 5.5 discusses Bayesian sampling plans that allow plans controlling producer's and consumer's risks to be designed while requiring only small sample sizes.

Operating Characteristic Curve (n=1, c=0) sampling plan

The ideal Operating Characteristic Curve, i.e. where analytical measurement uncertainty is negligible, for this plan is shown below – basically this says that if a percentage P of the product in a lot is nonconforming then the probability of acceptance, $p.acc = 1 - P$.

Figure 10 OC curve for (n=1, c=0) plan, negligible MU



This and the points above show that there is a high risk of making an incorrect assessment of a lot using these plans, and indeed any plan based on a small number of samples. This suggests that confirmatory sampling and testing using on a more discriminatory sampling plan should be undertaken prior to raising a dispute.

3.1.3 Example: Attributes plans based on AQL of 6.5%

The CXS 3-1981 Standard for Canned Salmon has three provisions based on sampling that must be met for acceptance of a lot.

Section 8 defines defective sampling units (cans) in terms of foreign matter, odour and flavour, texture, discolouration exceeding 5% of the net contents and objectionable matter.

A lot is accepted provided:

1. the total number of defectives as classified according to Section 8 does not exceed the acceptance number (c) of an appropriate sampling plan with an AQL of 6.5%;

2. the total number of sample units not meeting the form of presentation as defined in Section 2.3 does not exceed the acceptance number (c) of an appropriate sampling plan with an AQL of 6.5%;

3. the average net weight and the average drained weight where appropriate of all sample units examined is not less than the declared weight or drained weight as appropriate, and provided there is no unreasonable shortage in any individual container.

In this standard, Section 7.1 contains the following information:

SAMPLING	
(i)	Sampling of lots for examination of the final product as prescribed in Section 3.3 shall be in accordance with an appropriate sampling plan with an AQL of 6.5%.
(ii)	Sampling of lots for examination of net weight shall be carried out in accordance with an appropriate sampling plan meeting the criteria established by the CAC.

The following options for sampling plans are from ISO 2859-1999, CXG 50-2004 Appendix II ISO INSPECTION PLANS INDEXED BY PRODUCER'S RISK from ISO 2859-1 Plans indexed by AQL.

Using the table, for a lot of size N=500 cans, a sample size of n=50 cans would be required at the normal inspection level, with the lot rejected if more than 7 of those cans were nonconforming, that is if they contained foreign matter, objectionable matter, atypical odour or flavour, abnormal texture, or discolouration exceeding 5% of the net contents.

Presumably, in this plan, each of the 50 cans is examined for each of the listed defects with a can classified as nonconforming if the criterion is breached for any one of them.

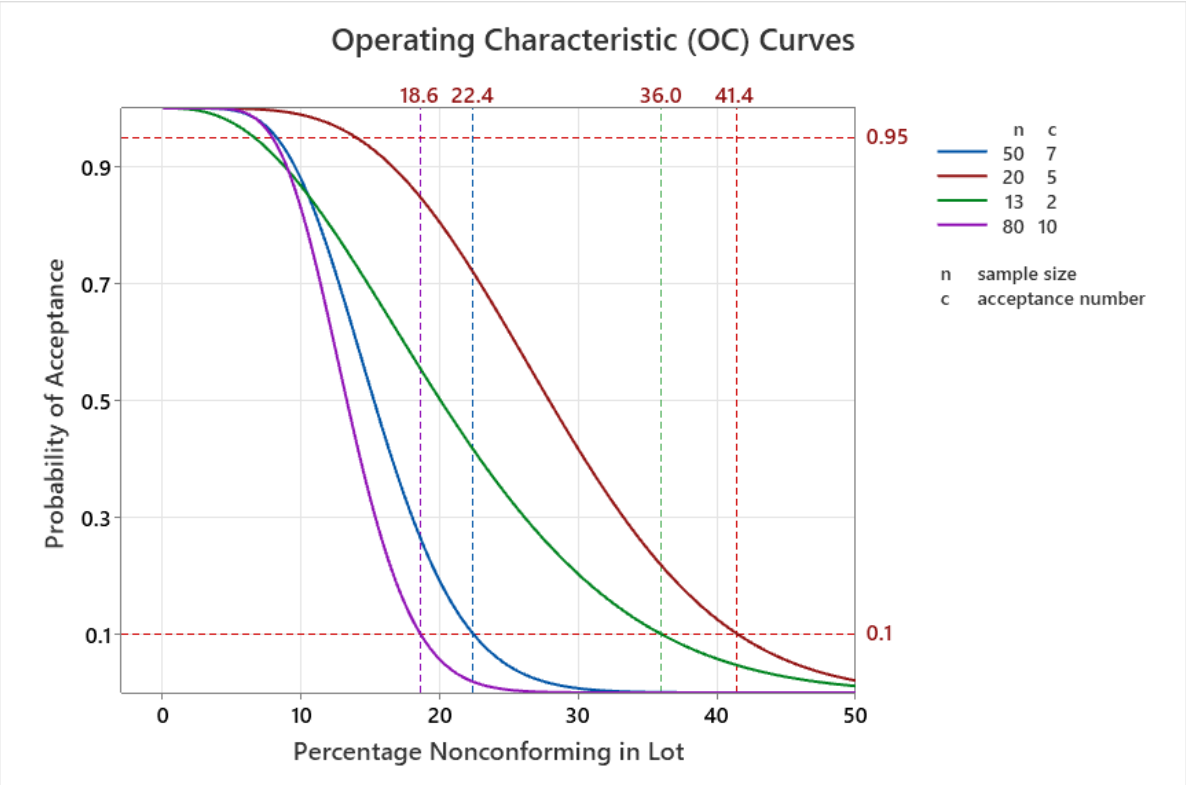
The consumer's risk quality level (CRQ) for this plan (n=50, c=7) is 22.4% meaning that there is a 10% chance of accepting a lot in which 22.4% of the cans are nonconforming. A decision should be made whether this is an acceptable level of risk before this plan is used.

This number of samples could be considered excessive, especially in terms of the overall lot size, although, as noted in CXG 50, the sampling fraction does not play a role in the design of sampling plans except possibly for quite small lots.

If it was decided to use the reduced inspection plan, (n=20, c=5) the consumer's risk quality level would increase to CRQ = 41.4%. If the sample size versus lot size relationship was disregarded and if the plan (n=13, c=2) was applied, then the CRQ would increase to 36%. On the other hand, if the plan (n = 80, c=10) was used, then the consumer's risk quantity level CRQ would decrease to 18.6%.

Operating characteristic curves for the four options are:

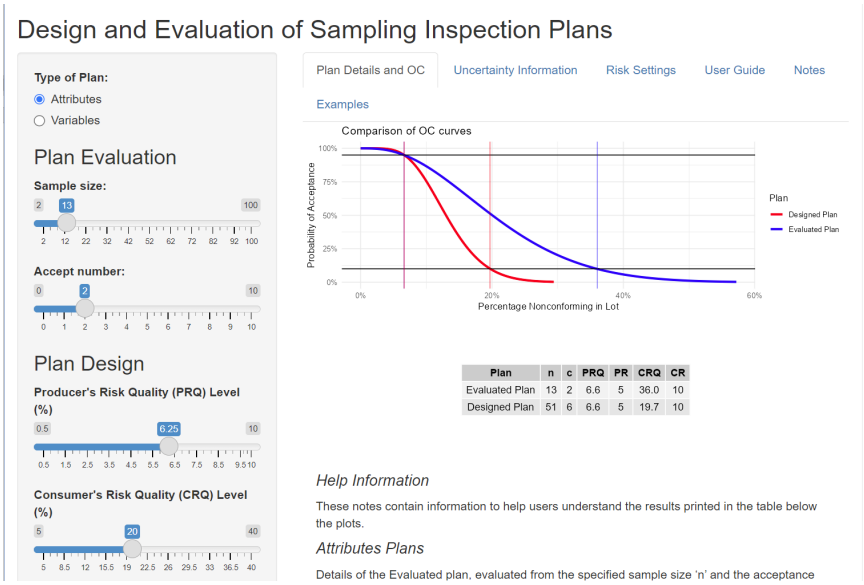
Figure 11 OC curves for ISO attribute plans



Extension

Suppose we wanted to modify this plan to provide a greater level of assurance to customers against accepting lots containing high levels of nonconforming product and that we were happy to keep the PRQ level at 6.5% but set the CRQ at 20%. This is easily accomplished using the app that shows the required plan is (n=50, c=6). The output from the app is shown below. Note that unlike the ISO tables, the plan does not depend on the lot size.

Figure 12 OC curve for modified plan based on ISO plan



3.2 Examples for Variables plans

3.2.1 Example: Variables plan with negligible MU

Fat in Whole milk powder

The examples for variables plans are based on the provision for fat in whole milk powder from Codex Standard CXS 207-1999. The provision states that the milkfat content should exceed 26%.

From Codex Standard CXS 234 the analytical test method for fat in whole milk powder is the Röse-Gottlieb method (ISO 23318/IDF 249). The method contains the following precision data:

The repeatability limit is 0.2% (0.2 percentage points)
The reproducibility limit is 0.3% (0.3 percentage points)

Using these values, the repeatability standard deviation is calculated as:

$$\sigma_r = 0.2/(1.96 \cdot \sqrt{2}) = 0.2/2.77 = 0.072\ (percentage\ points)$$

With a similar calculation for the reproducibility standard deviation:

$$\sigma_R = 0.3/\left(1.96 \cdot \sqrt{2}\right) = 0.3/2.77 = 0.108\ (percentage\ points)$$

Using these values, the 'between-laboratory' standard deviation is calculated as:

$$\sigma_L = \sqrt{\sigma_R^2 - \sigma_r^2} = \sqrt{0.108^2 - 0.072^2} = 0.081\ (percentage\ points)$$

Refer to the Guidelines on Analytical Terminology CAC/GL 72-2009 for more information about the repeatability and reproducibility.

Key steps in the step-by-step design process

The design process involves finding the number of samples n, and the acceptability constant k, in order that the risks of incorrect decisions can be controlled the levels specified by the producer's risk (PR) and consumer's risk (CR) at their corresponding quality levels PRQ and CRQ respectively.

In this context the plan evaluation feature of the app might not be relevant but still might be useful for comparison of the designed plan with other options.

1. Attributes or variables?

Fat is a measured characteristic so this is an example of variables data.

2. Does the provision relate to the average value or to the entire distribution, i.e. to a maximum or minimum allowable level for the characteristic in the lot?

The provision specifies a minimum limit so relates to the entire distribution – 'most' of the product in the lot should comply.

3. Does the distribution of the characteristic follow a normal or some other distribution?

For the purposes of this example, we assume that lot consists of 1000 cans of milk powder and that the milkfat content of the cans in the lot is normally distributed. This is a reasonable assumption if the cans are produced using powder from a manufacturing process in a state of statistical control.

4. Is measurement uncertainty negligible or non-negligible?

If the standard deviation representing the variation of the characteristic in the lot was $\sigma = 0.3$, then the error-variance ratio is $(0.072/0.3)^2 = 0.058$, that, being less than 10%, means it would not be necessary to allow for measurement uncertainty in the design of this plan.

5. Specify the stringency required for the sampling plan.

Consumer's Risk Quality (CRQ):

What of percentage nonconforming cans would you be prepared to allow in lots that you would want to reject most of the time?

Consumer's Risk (CR):

How often would you want to accept such lots? (Usually 10%)

Producer's Risk Quality (PRQ)

What percentage of nonconforming cans would you be prepared to allow in lots that you would want to accept most of the time?

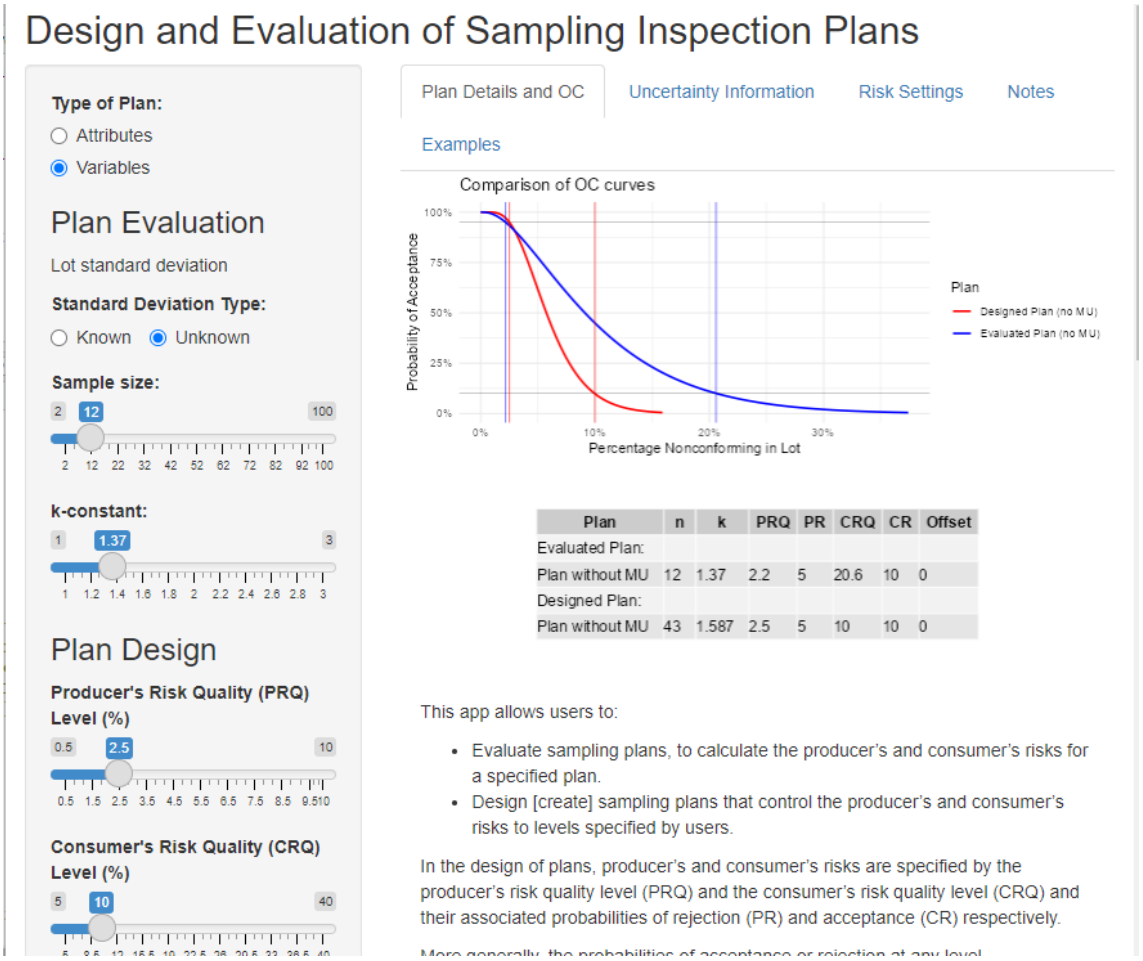
Consumer's Risk (CR):

How often would you want to reject such lots? (Usually 5%)

In this example the CRQ was chosen as 10% and the PRQ as 2.5%, with the CR and PR unchanged. This means that the plan will have:

- A 10% chance of accepting a lot in which 10% of the product is nonconforming.
- A 5% chance of rejecting a lot in which 2.5% of the product is nonconforming.

Figure 13 OC curve for variables plan - negligible measurement uncertainty



The plan required to control risks to the specified levels is (n=43, k=1.59), i.e. 43 samples need to be taken from the lot and tested. The lot is accepted provided the average and the standard deviation of the results meet the acceptance criterion:

$$\bar{x} - \mathrm{k} \times s \geq 26$$

where:

- $\bar{x}$ is the average of the 43 individual results and 's' their standard deviation,
- *k* is the acceptability constant, k=1.59 in this example.
- It is assumed that the measurements are expressed as percentages e.g. moisture of 5% on a weight/weight basis.

Note that the ISO plan for PRQ = 2.5% for a lot size N=1000 (Sample Code J, Inspection Level II) is (n=12, k=1.370) if the lot standard deviation is known and (n=46, k=1.482) if the lot standard deviation is unknown. Both plans have an actual PRQ of about 3.4% and a CRQ of about 11.3%.

Negligible measurement uncertainty

The validity of the assumption of negligible measurement uncertainty can be checked using the app, as follows.

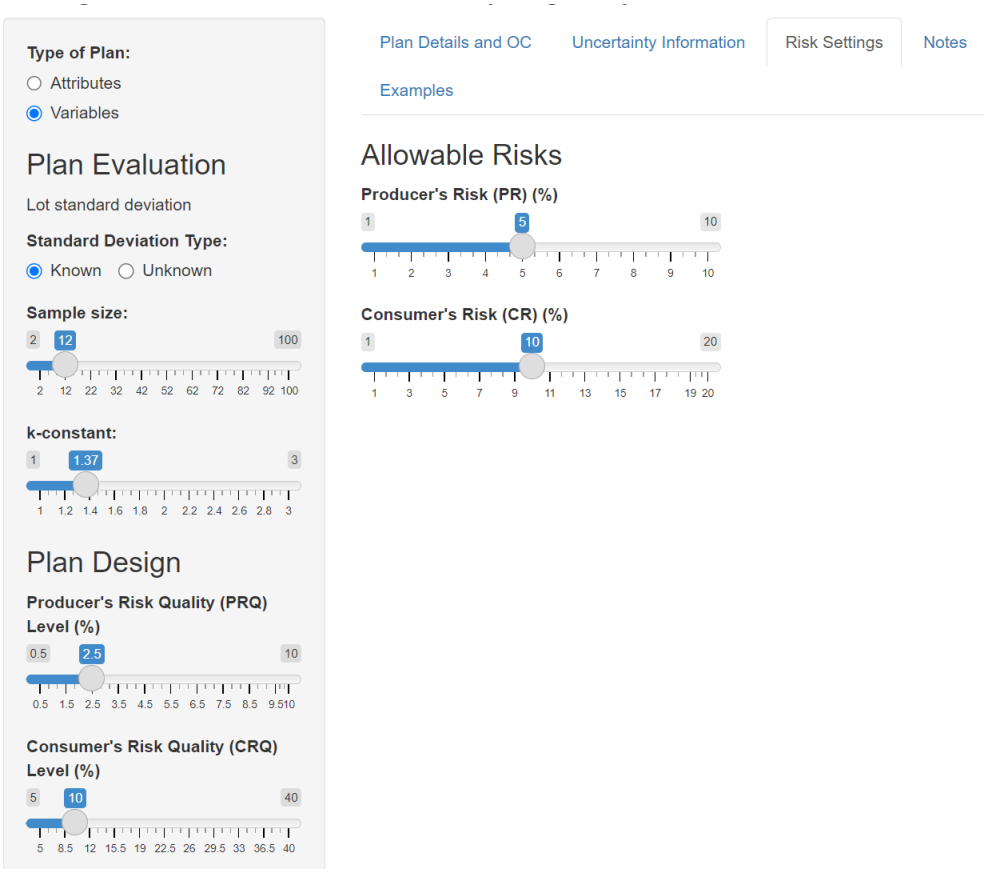
Step 1.

Open the app and select 'Variables' as the type of plan.

Step 2.

In the Plan Design section of the Plan Details and OC tab, set the Producer's Risk Quality level (PRQ) to 2.5% and the Consumer's Risk Quality level (CRQ) to 10%. There is no need to change the producer's and consumer's risks from their default settings, 5% and 10% respectively, at least for this example.

Figure 14 Negligible MU example - setting allowable risks



Step 3

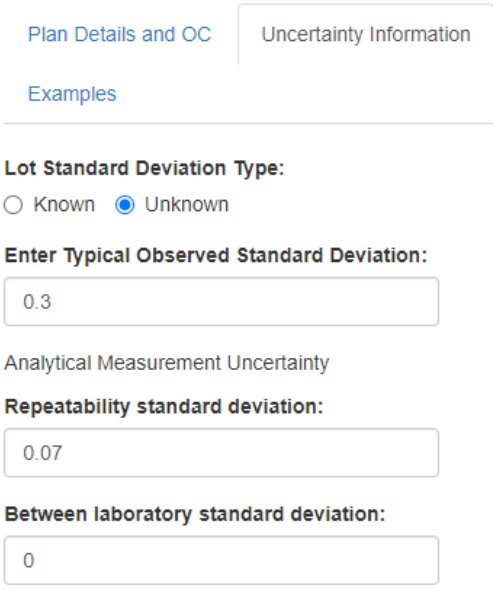
Go to the Uncertainty Information tab.

Select the “Lot Standard Deviation Type” to “Unknown”.

In the same window set the typical value of the observed standard deviation to 0.30 using the spin button ( ) or by entering the value directly and the repeatability standard deviation to $\sigma_r = 0.07$ similarly.

Note that the app has been designed so that if the lot standard deviation is known that value is used but if it is unknown, a typical value of the observed total standard deviation inclusive of repeatability error is entered.

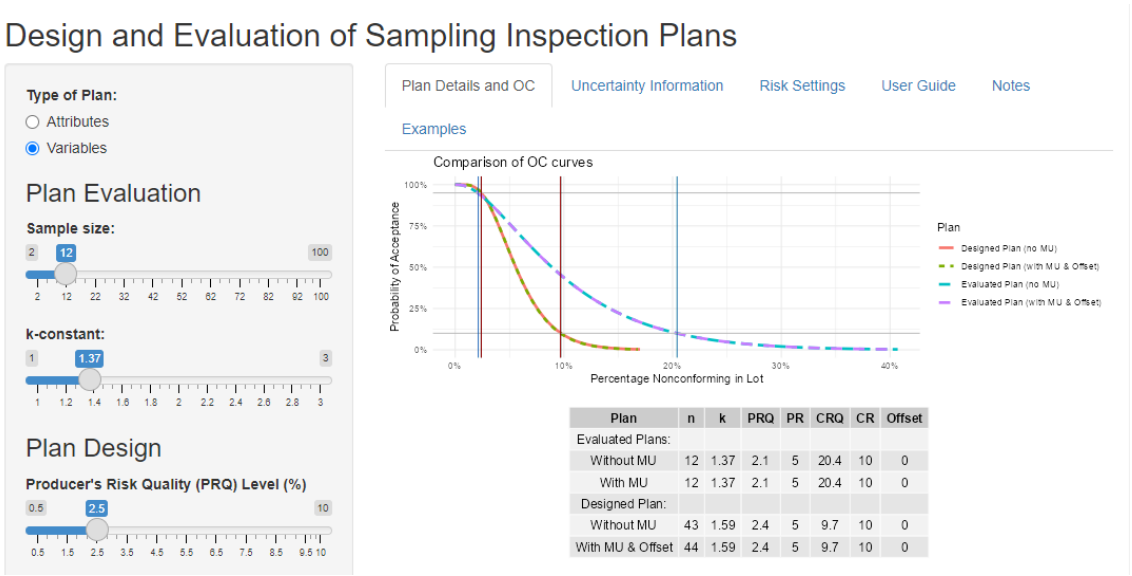
Figure 15 Variables Plan example - setting uncertainty information



Step 4

Return to the Plan Details and OC tab to view details of the plan and the OC curve. The app shows the OC curves and details of the plans including the risks,

Figure 16 Variables plan example - output



Plan	n	k	PRQ	PR	CRQ	CR	Offset
Evaluated Plans:							
Without MU	12	1.37	2.1	5	20.4	10	0
With MU	12	1.37	2.1	5	20.4	10	0
Designed Plan:							
Without MU	43	1.59	2.4	5	9.7	10	0
With MU & Offset	44	1.59	2.4	5	9.7	10	0

The last row of the table shows that the designed plan with the specified risks, allowing for the measurement uncertainty specified is (n=44, k=1. 59), the same as the plan where there is no measurement uncertainty shown in the line above. This differs slightly from the evaluated plan with the same n and k due to rounding of the k value within the app.

Notice also that although less than 10%, the error variance ratio of 0.058 means that one would expect the sample number to increase from 43 to $43 \times (1 + 0.058) = 45.5$ that, when rounded up gives n=46. However, for the unknown lot standard deviation case Hahn's adjustment (CXG 50 section 5.2.7) has been applied, meaning that although allowance must be made for the unknown standard deviation, it is not necessary to adjust the lot standard deviation for the measurement uncertainty. In any case, there is little difference between the plans in their sample numbers or risks and the 'error-free' plan can be used.

The lot is accepted provided the average and the standard deviation of the results meet the acceptance criterion:

$$\bar{x} - 1.59 \times s \geq 26$$

where:

- $\bar{x}$ is the average of the 43 individual results and 's' their standard deviation.
- It is assumed that the measurements are expressed as percentages e.g. a fat level of 26.5% on a weight/weight basis.

Although it is unnecessary in this example, the calculations assume that Hahn's adjustment has been applied to the observed total standard deviation, calculated from the inspection data. The adjusted standard deviation can be calculated using the formula:

$$s_{adj}^2 = s_{obs}^2 - \sigma_r^2$$

provided the right-hand side is greater than zero, otherwise the value of the adjusted standard deviation is taken as zero.

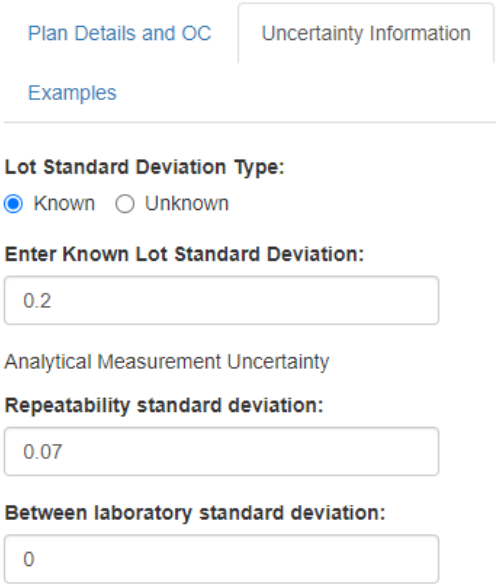
3.2.2 Example: Variables plan with non-negligible MU with no laboratory bias

It is assumed that the specified producer's and consumer's risks are set at the same levels and the between laboratory component of the analytical measurement uncertainty is negligible. However, in this example the lot standard deviation is assumed known.

If the lot standard deviation was $\sigma = 0.2$, the error-variance ratio is $(0.072/0.2)^2 = 0.13$ and, being greater than 10%, suggests that to allow for measurement uncertainty the number of samples should be increased to $19 \times (1 + 0.13) = 21.5 = 22$ after rounding.

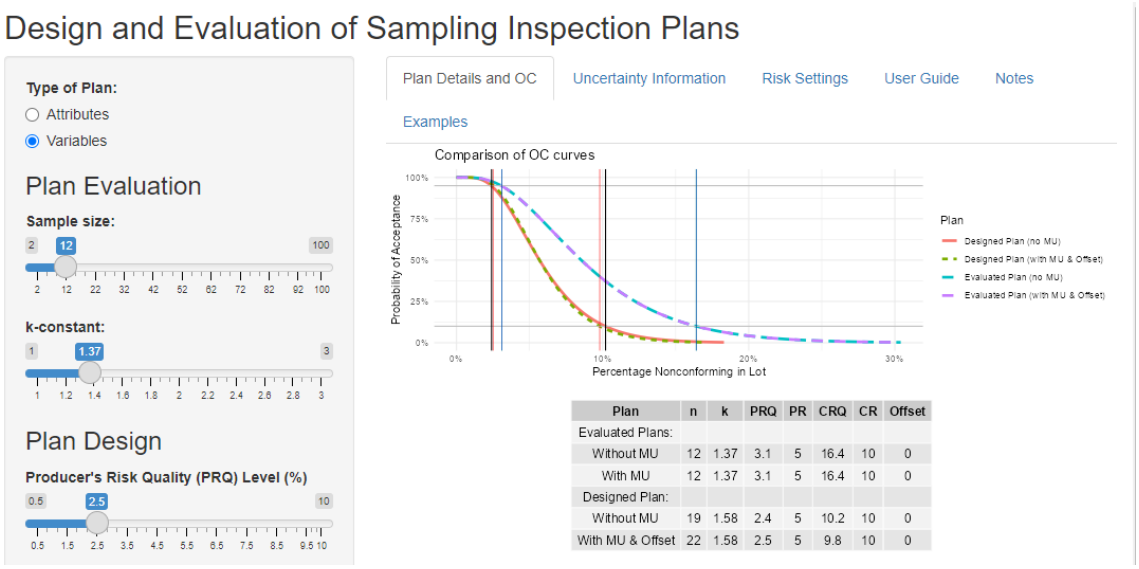
The app is used in the same way as in the example above, except that the lot standard deviation type is set to known (and, of course, a different value of the lot standard deviation is entered).

Figure 17 Variables plan example - non-negligible MU



The last row of the table below the OC curves confirm the calculation above, that there is a modest increase in sample size to n=22.

Figure 18 Variables Plan design - non-negligible MU



Alternatively, in the known standard deviation case, or if the error variance ratio γ is known, say from a measurement error study if the lot standard deviation is unknown, the acceptability constant k can be reduced to compensate for the increase in variability without the need to increase the sample size:

$$k^* = k/\sqrt{1+\gamma}$$

where:

k is the acceptability constant for the original plan,

k* is the acceptability constant for the modified plan.

Suppose the variables sampling plan (n=23, k=1.19) is being used to assess compliance of a particular characteristic having an upper limit of U = 10 and we have obtained test results as follows:

9.92, 9.85, 10, 9.62, 9.94, 10.02, 9.87, 9.8, 9.87, 9.95, 10.05, 10.03, 9.57, 9.83, 9.93, 9.93, 9.89, 9.79, 9.97, 9.96, 9.92, 9.83, 10.05

It is known from a previous measurement study that the error-variance ratio, the ratio of the repeatability variance to the lot standard deviation variance, is 0.25. Recall from CXG 50-2004 that the variance is the square of the standard deviation.

If the assessment of compliance proceeds in the usual way, the mean value of the results is m=9.90, the standard deviation s = 0.12, so that the acceptance criterion has a value of 9.90 + 1.19*0.12 = 10.04, and the lot should not be accepted.

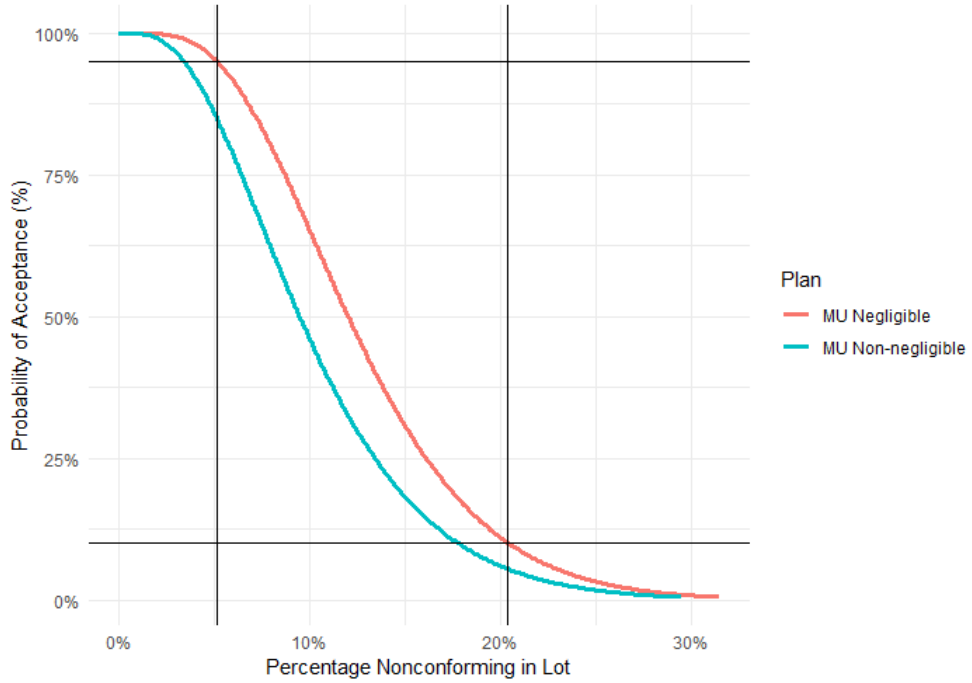
However, it is possible that measurement uncertainty has caused the lot to fail inspection. Hahn's adjustment can be applied to adjust the observed standard deviation for measurement uncertainty using the repeatability standard deviation is known from method validation. Supposing the repeatability standard deviation is $\sigma_e = 0.10$, the adjusted observed standard deviation s_{adj} is calculated by:

$$s_{adj}^2 = s_{obs}^2 - s_e^2 = 0.12^2 - 0.10^2 = 0.0044$$

so that the adjusted standard deviation $s_{adj} = 0.066$ and the updated value of the acceptance criterion is 9.90+1.19*0.066 = 9.98 and the lot can be accepted.

The OC curves below show that the probability of acceptance at any given percentage nonconforming in a lot will be less when repeatability-type measurement uncertainty is present.

Figure 19 OC curves with and without MU (no bias)



Plan	n	k	PRQ	PR	CRQ	CR
Error-free Plan	23	1.19	5.1	5	20.4	10
Error-prone Plan	23	1.19	3.4	5	17.8	10

Another way of overcoming non-negligible, repeatability measurement uncertainty is to increase the sample size; ISO3951-1: 2013 gives the formula:

$$n^* = n(1+\gamma)$$

where:

n is the sample size for the original plan in which measurement uncertainty is negligible,

n* is the sample size for the modified plan, and

γ is the error-variance ratio.

Alternatively, if the error variance ratio γ was known, the acceptability constant k can be reduced to compensate for the increased variability without the need to increase the sample size:

$$k^* = k/\sqrt{1+\gamma}$$

where:

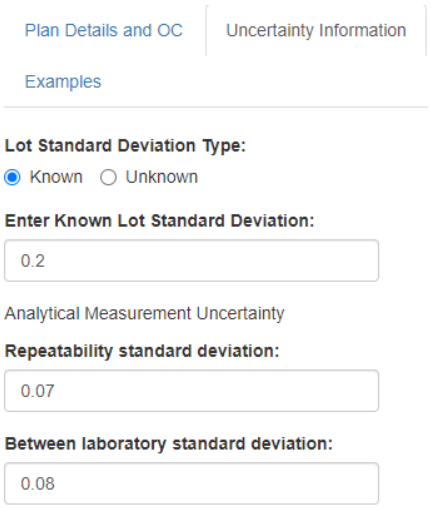
k is the acceptability constant for the original plan,

k* is the acceptability constant for the modified plan.

3.2.3 Example: Variables plan with non-negligible MU with laboratory bias

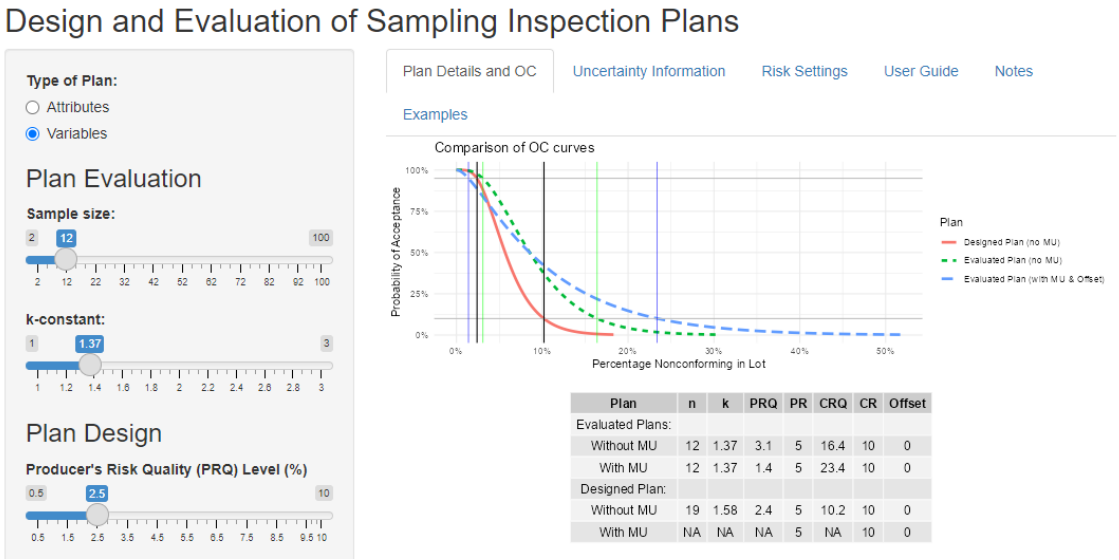
In this example it is assumed that the lot standard deviation of σ = 0.2 is known and that the between laboratory component of measurement uncertainty has a standard deviation of $\sigma_L = 0.08$ as in the original example (Section 3.2.1).

Figure 20 Entering uncertainty information - Non-negligible MU (inc. bias)



The NA (not available) values in last row of the table shows that a plan allowing control of the producer's and consumer's risks to the levels specified cannot be found.

Figure 21 Output - Non-negligible MU example



Plan	n	k	PRQ	PR	CRQ	CR	Offset
Evaluated Plans:							
Without MU	12	1.37	3.1	5	16.4	10	0
With MU	12	1.37	1.4	5	23.4	10	0
Designed Plan:							
Without MU	19	1.58	2.4	5	10.2	10	0
With MU	NA	NA	NA	5	NA	10	0

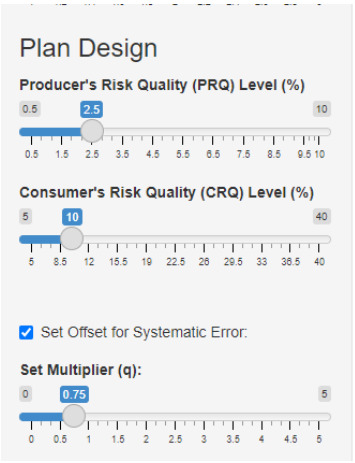
To find a plan it is necessary to introduce an offset.

Open the sub-window "Set Offset for Systematic Error" and move the slider to set a value of "q" until a plan is found. For example, as shown in the image below, if the multiplier is set to q = 0.75, then the offset in the acceptance criterion is then q*sigma_L = 0.75*0.08 = 0.06 so that with n=19 and k = 1.58 so the acceptance criterion would then become:

$$\bar{x} + k \cdot \sigma + 0.06 \leq USL$$

Note that while the consumer's risk quality level (CRQ) remains unchanged, the producer's risk quality is significantly reduced.

Figure 22 Plan details - non-negligible MU example



Plan	n	k	PRQ	PR	CRQ	CR	Offset
Evaluated Plans:							
Without MU	12	1.37	3.1	5	16.4	10	0
With MU	12	1.37	0.6	5	15.3	10	0.06
Designed Plan:							
Without MU	19	1.58	2.4	5	10.2	10	0
With MU & Offset	19	1.58	0.4	5	10	10	0.06

Help Information

These notes contain information to help users understand the results printed in the table below the plots.

Attributes Plans

Details of the Evaluated plan, evaluated from the specified sample size 'n' and the acceptance number 'c' and the designed plan, based on the specified Producer's and Consumer's risks are shown.

Variables Plans

Details of the Evaluated plans, evaluated from the specified sample size 'n' and the acceptability constant 'k' are shown. The OC curves for these plans are shown both without and with the effect of measurement uncertainty,

Details of the Designed, designed from the specified Producer's and Consumer's risks are shown. The OC

3.2.4 Example: Fractional Nonconformance plans

Suppose we have measurements from testing 15 samples from a lot to assess whether the lot conforms with the lower specification limit of L = 50. The measurement process is known to be normally distributed, with no laboratory bias and a standard deviation of $\sigma = 0.045$.

The following results were obtained:

50.01, 50.04, 50.07, 50.1, 50.15, 50.2, 50.29, 50.42, 50.45, 50.48, 50.55, 50.6, 50.8, 51.2, 51.3

The fractional nonconformance values for each sample can be calculated using Excel, using the formula:

$$fnc = NORMDIST(50, x, 0.045, TRUE)$$

where 'x' represents a single test result. This gives the following FNC values:

0.4121, 0.187, 0.0599, 0.0131, 0.0004, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0

The sum of these values is 0.6725, so if the acceptance limit Ac was 0.75 the lot would be accepted.

This example shows the principle behind the calculation, that can easily be extended to allow for measurement uncertainty distributions other than the normal distribution.

The following shows the OC curves for a variables plan and for a FNC plan, both with the same error-variance ratio.

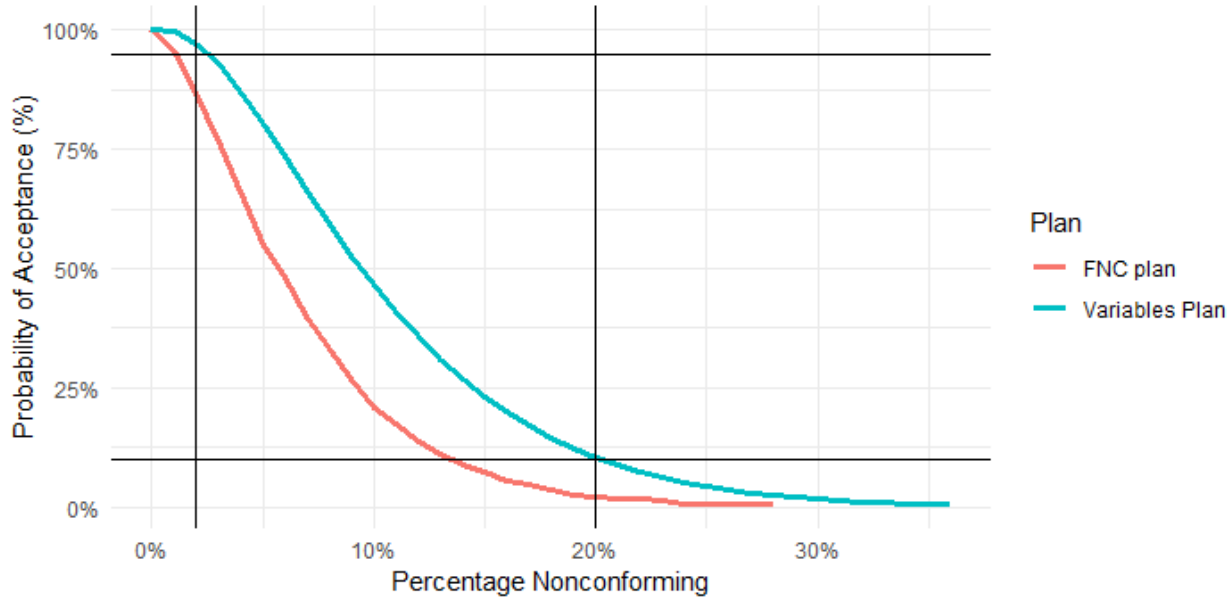
Fractional nonconformance plans are an example of attribute-variables (attri-variables) plans where measurements are reclassified into another 'measure' of conformance for each sample, with the decision on acceptance of the lot made using the sum of these new measures.

The ICMSF sampling plans used in microbiological assessments for counted characteristics are another example of attri-variables plans as are attributes plans used with measurements that are classified as pass or fail with respect to a limit.

See also Section 4.4.

The FNC app uses a modified procedure from that described above; differences of each observation from the sample mean are also taken into account. This provides a more stringent procedure requiring fewer samples than the unmodified method. Both methods are described in Govindaraju, K. and Jones, G. [31].

Figure 23 Fractional Nonconformance Plans



Plan	n	k_Ac	AQL	AQL.risk	LQL	LQL.risk
Variables Plan	15	1.20	2	5	20	10
FNC plan	15	0.75	1	5	14	10

3.3 Lots consisting of bulk materials

3.3.1 Example: Aflatoxin sampling plans due to Whitaker (2006) et al.

Refer to 5.6.5 Aflatoxin sampling plans

Shelled Almonds for further processing

Suppose the average concentration of aflatoxins in the lot was C = 8 µg/kg and ns = 20000, 20 kg @ 1000 shelled nuts per kg, were taken as a sample, and this sample was ground and well-mixed composite formed. If a subsample of 50g was taken and a single aliquot (n_a=1) tested, the standard deviation S representing the uncertainty of the average level would be:

$$S^{2=}\frac{7730 \times 5.759}{20 \times 1000}8^{1.561} + \frac{100 \times 0.170}{50}8^{1.646} + \frac{0.048}{1}8^{2} = 70.67$$

Giving S = 8.41. The first component representing the sample-by-sample variation is much larger than the other two.

The maximum limit for shelled nuts for further processing is 20 µg/kg, based on an initial sample of 20kg shelled almonds and one laboratory determination.

At an average level of contamination of C=8 µg/kg, the variance $S^2 = 70.67$ and from the formula above, the value of k is worked out using:

$$70.67 = 8 + 8 \times 8/k$$

from which

$$k = \frac{64}{70.67 - 8} = 1.0212 \; and \; \frac{k}{C+k} = \frac{1.0212}{8 + 1.0212} = 0.1132$$

The probability of acceptance be calculated using Excel:

BETA.DIST(k/(C+k),k,maximum_Limit,TRUE) that is equivalent to the Negative Binomial distribution[3].

$$BETA.DIST\left(\frac{k}{C+k}, k, maximum.limit, TRUE\right) = BETA.DIST(0.1132,1.0212,20,TRUE) = 0.906$$

or 90.6%

[3] Although the negative binomial distribution function is available in Excel, it is not in a form suitable for these calculations.

Note that the probability of acceptance at the maximum limit C = 20µg/kg is 0.622, that shows again that the principle of offsets has been employed in the setting of limits to provide consumer protection.

The calculations of the probabilities of acceptance in the Mycotoxin S&T Guide appear approximate, the actual calculations are unknown but the differences from results calculated in other known ways are small enough so as not to matter.

Probabilities of Acceptance for Shelled Corn (n_s=3000, n_{ss}=50, n_a = 1)

Concentration (µg/g)	Variance	Mycotoxin S&T Guide (%)	Negative Binomial (R) (%)	Beta Distribution (R) (%)	Beta Distribution (Excel) (%)
0	0	100	100	100	100
5	72.76	94.07	94.29	94.29	94.29
10	148.01	84.9	85.3	85.3	85.3
20	302.74	61.53	62.23	62.23	62.23
30	461.41	38.87	39.8	39.8	39.80

3.3.2 Example: Plans based on the beta distribution

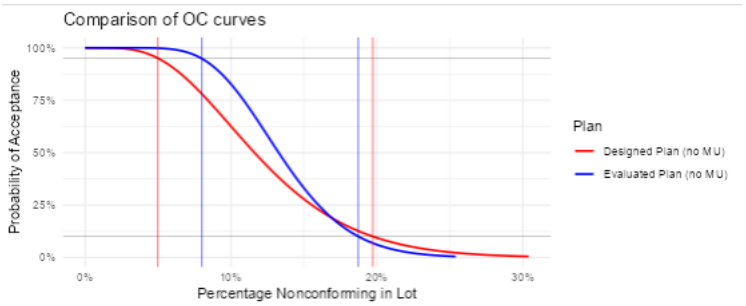
Plan for Capsaicin – based on Codex Standard 294-2023

Codex Standard CXS294 - 2023 for Gochujang contains a provision for capsaicin, that the levels should not be less than 10 mg/kg on a weight:weight (w/w) basis with lot acceptance decided using an attributes sampling plan with AQL = 6.5%, where a container is classed as nonconforming if the result from testing a sample taken from that container is less than the limit.

The number of samples will depend on the size of the lot but could be considerable, e.g. n=80 samples for a lot consisting of 1000 containers [packages]. However, capsaicin is tested using the HPLC method, so it is not feasible to perform more than relatively few tests on each lot.

As commented elsewhere, the use of attributes plans classifying measurements as attributes is inefficient and, for a lot size of 1000 containers the corresponding variables plan from ISO3951-1 (standard deviation unknown, negligible measurement uncertainty) with AQL = 6.5% is n=52, k=1.120, that has an AQL (PRQ) of 8%, with PR = 5%, and LQL (CRQ) of 18.7%, with CR = 10%.

Figure 24 Capsaicin example - ISO plans

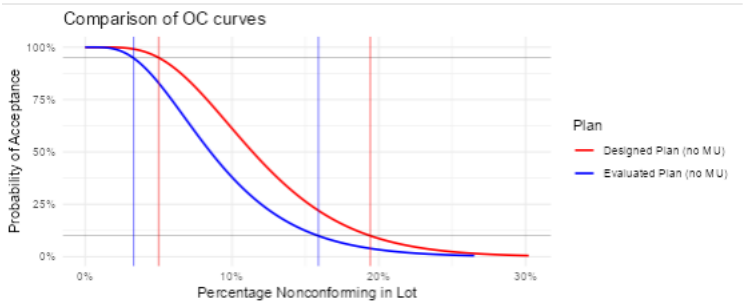


Plan	n	k	PRQ	PR	CRQ	CR	Offset
Evaluated Plan:							
Plan without MU	52	1.12	8	5	18.7	10	0
Designed Plan:							
Plan without MU	24	1.21	5	5	19.7	10	0

An alternative approach is to consider compliance of capsaicin levels in the overall lot rather than at the individual container level and, being a measured characteristic, it means that variables plans could be used.

Using the same consumer's and producer's risks as those for protein and moisture above (a producer's risk of 5% of rejecting lots containing 5% nonconforming product and a consumer's risk of 10% of accepting lots containing 20% nonconforming product) the resulting variables plan is (n=14, k=1.205) assuming the lot standard deviation is known. This plan can be modified to allow for non-negligible measurement uncertainty.

Figure 25 Capsaicin example - Variables plan



Plan	n	k	PRQ	PR	CRQ	CR	Offset
Evaluated Plan:							
Plan without MU	12	1.37	3.3	5	15.9	10	0
Designed Plan:							
Plan without MU	14	1.205	5	5	19.4	10	0

Further, capsaicin is a compositional characteristic, so that, if measurement uncertainty is negligible plans based on the beta distribution (refer to CXG 50-2004 Section 4.3.1) would be applicable. Use of these plans would mean that:

(1) a composite sample is formed from a requisite number of subsamples, that number being determined in the design of the plan based on specifications of allowable risks.

Acceptance of the lot would be determined by an acceptance criterion of the form:

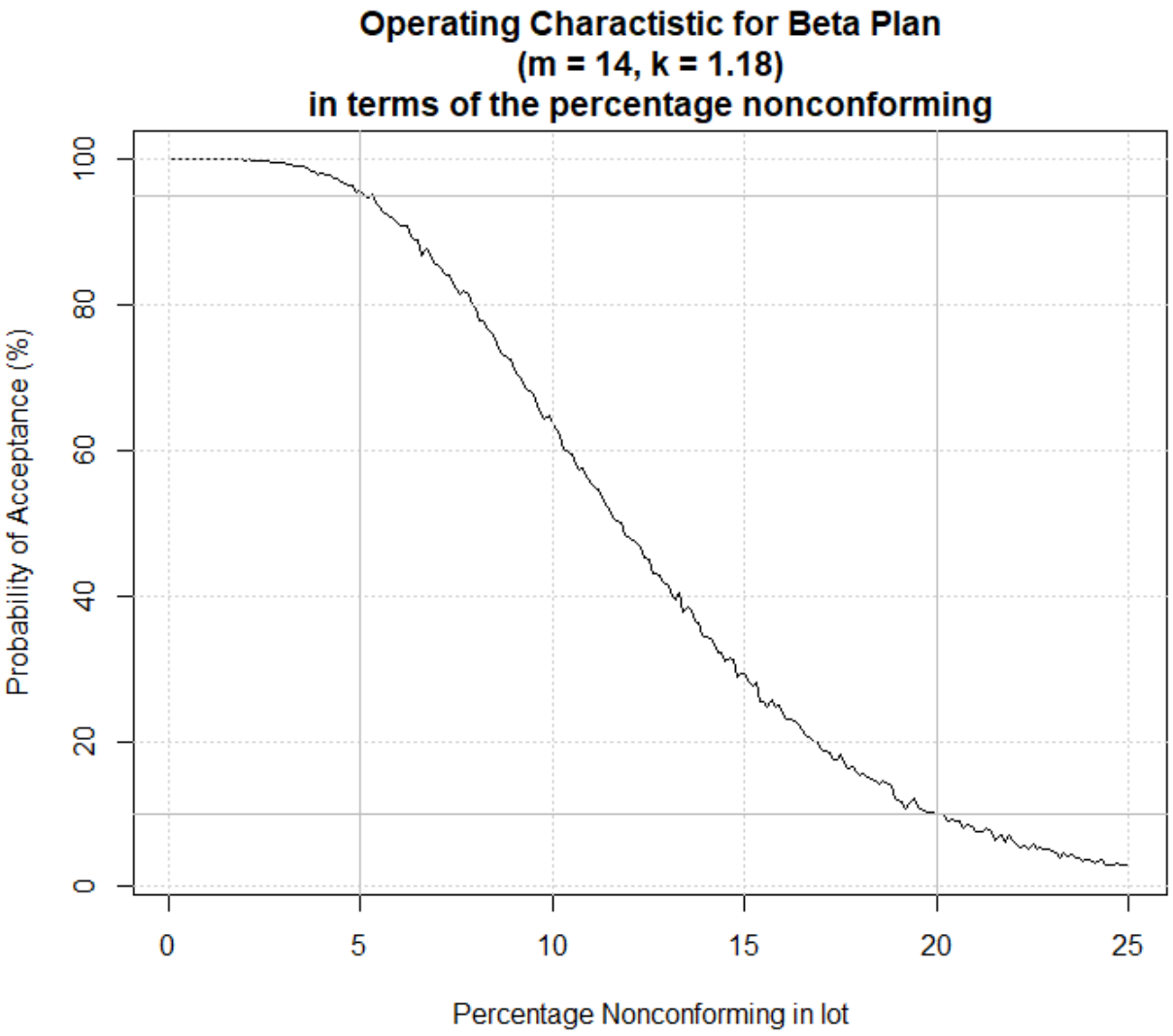
$P - k \times s \geq L$ where P is the test result or average test result and $s = \sqrt{P(1-P)/\theta}$, L is the minimum limit (10ppm) and k is the acceptability constant for the plan.

Historical data would first need to be analysed to estimate the precision parameter θ but a hypothetical value of the precision parameter of $\theta = 44 \times 10^6$ has been used in the following example.

Using those same consumer's and producer's risks the resulting plan is (m=14, k=1.18) i.e. a composite sample would be formed from 14 subsamples taken randomly from the lot, with the composite tested just once – the test result would then be the estimate of "P".

The Operating Characteristic for this plan is shown below.

Figure 26 Capsaicin example - Beta distribution plan



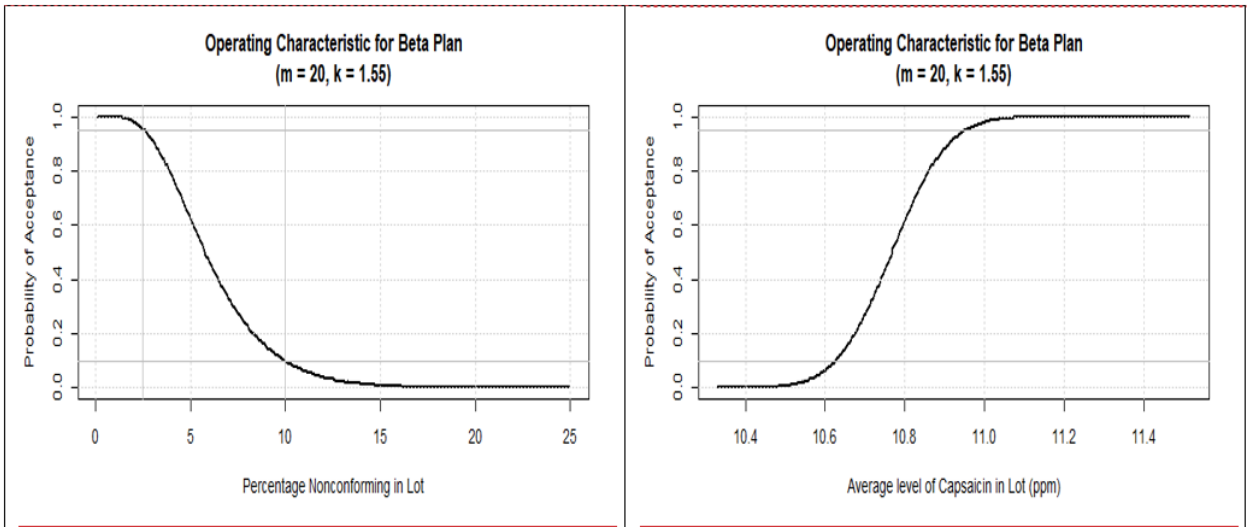
If however, we decided that capsaicin was a more critical characteristic for the product then we may wish to reduce the consumer's risk – instead of decreasing the chance of acceptance at the CRQ we can reduce the CRQ itself, to 10%, and also reducing the PRQ to 2.5%.

Consumer's Risk Quality level (CRQ)	
What percentage nonconforming would you allow in lots that you would want to reject most of the time?	10%
How often would you want to accept such lots (default = 10%)?	10%
Producer's Risk Quality level (PRQ)	
What percentage nonconforming would need to be present in lots that you would want to accept most of the time?	2.5%
How often would you want to reject such lots (default = 5%)?	5%

The corresponding sampling plan is (m=20, k=1.55) i.e. a composite sample would be formed from 20 subsamples randomly taken from the lot and the acceptance criterion would use a multiplier of the standard deviation of k=1.55.

The Operating Characteristics for this plan are shown below, in terms of both the percentage nonconforming and the average level of capsaicin in the lot.

Figure 27 OC curves for plan based on the beta distribution



3.4 Other sampling plans

3.4.1 Example: ISO sampling plans – 6.5% AQL

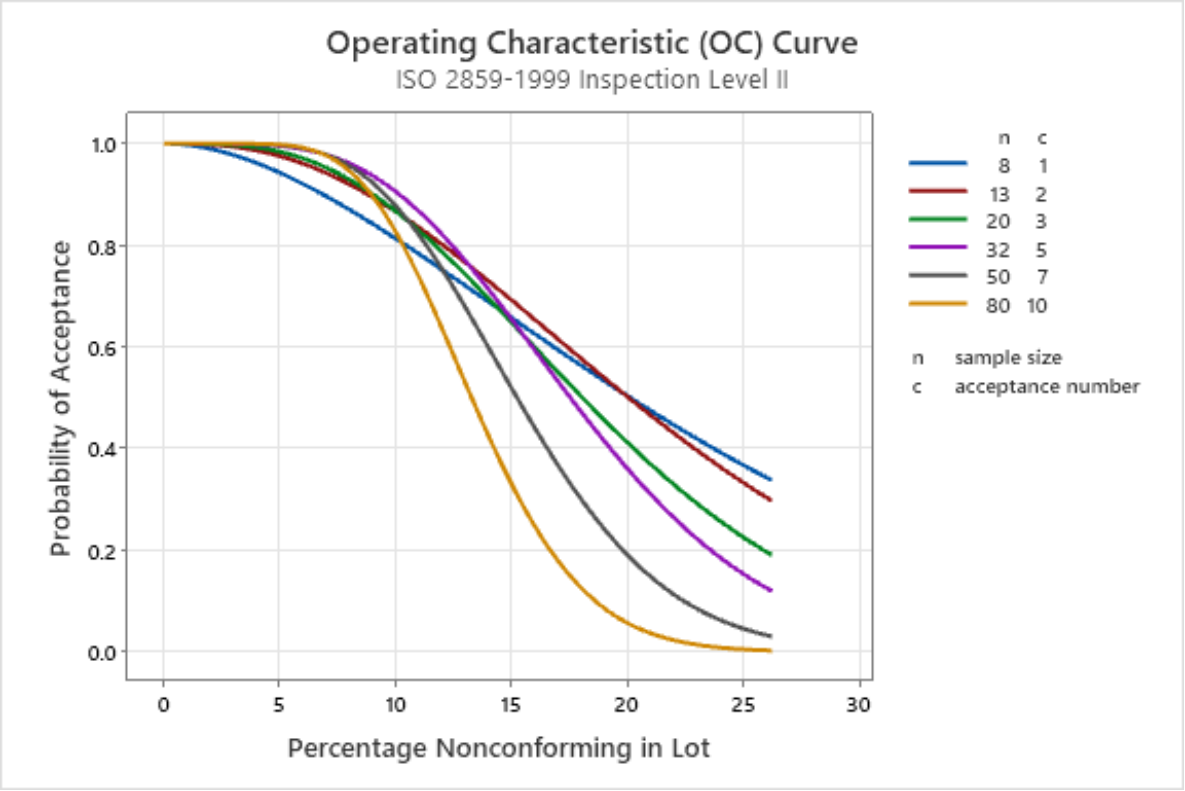
Refer to 3.2.2 Design of sampling plans, Table: Attributes plans from ISO 2859-1 for PRQ = 6.5 percent

A number of Codex standards contain sampling plans from the ISO standard ISO 2859[4] with an AQL of 6.5%, apparently because these plans were promulgated in the now defunct CODEX STAN 233, CODEX SAMPLING PLANS FOR PREPACKAGED FOODS (AQL 6.5) CODEX STAN 233-1969. While these plans might be suitable in some applications, users should first check that they will meet expectations around the control of producer's and consumer's risks before use. In particular, these plans might suffer from following problems:

- There could be poor control of the consumer's risk, that will vary according to the lot size.
- The plans should not be used by classifying variables data as attributes, except as a last resort, and not used in cases where measurement uncertainty is non-negligible.
- The ISO plans are intended to be used with switching rules; if not there will be no safeguards against deteriorating quality, nor any reward by way of reduced inspection for good quality.

[4] International standard ISO 2859-1: Sampling procedures for inspection by attributes - Part 1: Sampling schemes indexed by acceptance quality limit (AQL) for lot-by-lot inspection.

Figure 28 OC curve ISO 2859 attributes plans



From CXG 50 Appendix II ISO INSPECTION PLANS INDEXED BY PRODUCER'S RISK:

		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
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

		Inspection level					
Lot size	**AQL**	**reduced**		**normal**		**tightened**	
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

Producer's and Consumer's Risk Quality Levels (PR = 5%, CR = 10%)

n	c	PRQ%	CRQ%
8	1	4.64	40.62
13	2	6.60	35.98
20	3	7.14	30.42
32	5	8.50	27.07
50	7	8.22	22.42
80	10	7.91	18.60

Conclusion

Whilst possibly facilitating trade, plans with low sample numbers do not provide high levels of consumer protection, that will vary according to the lot size.

Net weight

It appears Codex has not provided any guidance on sampling plans for net weight. However, the need for provisions relating to weight has possibly been superseded by the introduction of weight legislation using the Average Quantity System, based on the OIML International Recommendation R087 published by BIPM.

3.4.2 *Ad hoc* plans

Suppose, for example, that 4-6 samples are taken, formed into a composite sample and a single laboratory sample taken from the thoroughly mixed composite for analysis.

This is not the standard statistical approach to the design of sampling plans as the plan is not designed from specifications of allowable risks; therefore, we must evaluate it to check that it will control risks satisfactorily.

Four options have been evaluated:

(1) use of the single result in an (n=1, c=0) sampling plan for the assessment of compliance to an average level (see 3.4.2.1 Compliance of the average level scenario evaluation),

(2) use in an (n=1, c=0) attributes plan (see 3.4.2.2 Compliance of the average level scenario evaluation),

(3) use in a variables plan (see 3.4.2.3 Variable Plan scenario evaluation) and

(4) use in a plan based on the beta distribution if the characteristic is a compositional proportion and measurement uncertainty is negligible (see 3.4.2.4. Beta distribution plan scenario evaluation).

Notation:

U the upper specification limit,

σ (*sigma*) the assumed known value of the lot standard deviation (rather than an estimate of σ).

Alternatively, the error-variance ratio must be well known; in this case it refers to the ratio of the reproducibility variance to the lot variance.

$$Error - variance\ ratio = \frac{u^2}{\sigma^2}$$

u the assumed known standard deviation representing the standard measurement uncertainty.

The uncertainty of the average level $\bar{x}$ of the composite sample formed by taking n samples will be $\sigma/\sqrt{n}$ and the uncertainty variance of the measured value will be $\sqrt{u^2 + \frac{\sigma^2}{n}}$

The following examples reinforce the guidance in ISO2859 that ad hoc sampling methods are not recommended since they lead to uncalculated risks and often to unjustifiably high risks; further, there is no logical basis for either the acceptance or rejection of the product.

3.4.2.1 Compliance of the average level scenario evaluation

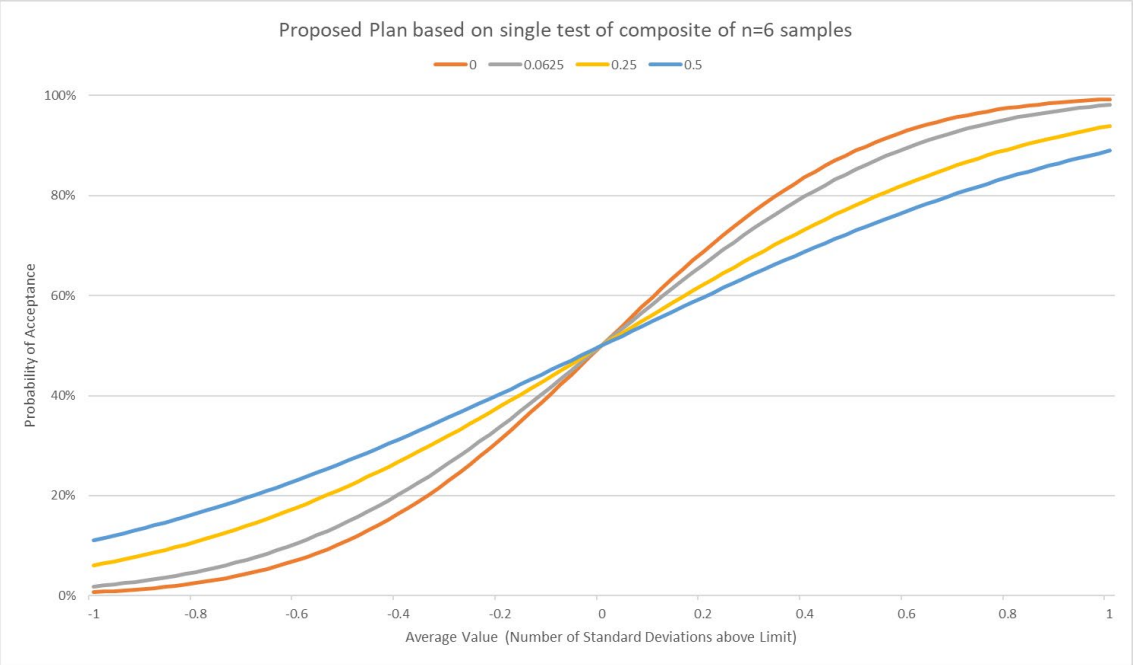
The probability of acceptance, of accepting a lot against a lower limit L in terms of the true average level μ in the lot is given by:

$$p.acc = NORMSDIST(k * sigma/SQRT(sigma\text{^}2/n + u\text{^}2))$$

Using Excel formula notation, where:

k*sigma is the offset from the limit.

Figure 29 OC curves - ad hoc plans - complinace of average level



The different lines show the OC curves for different values of the error-variance ratio.

Conclusion

This shows that the proposed plan is ineffective for assessing compliance of an average level – for example there is still a high chance of acceptance when the true average level is a reasonable number of standard deviations below the limit; for example, around 20% chance of acceptance when the average level is 0.5 standard deviations below.

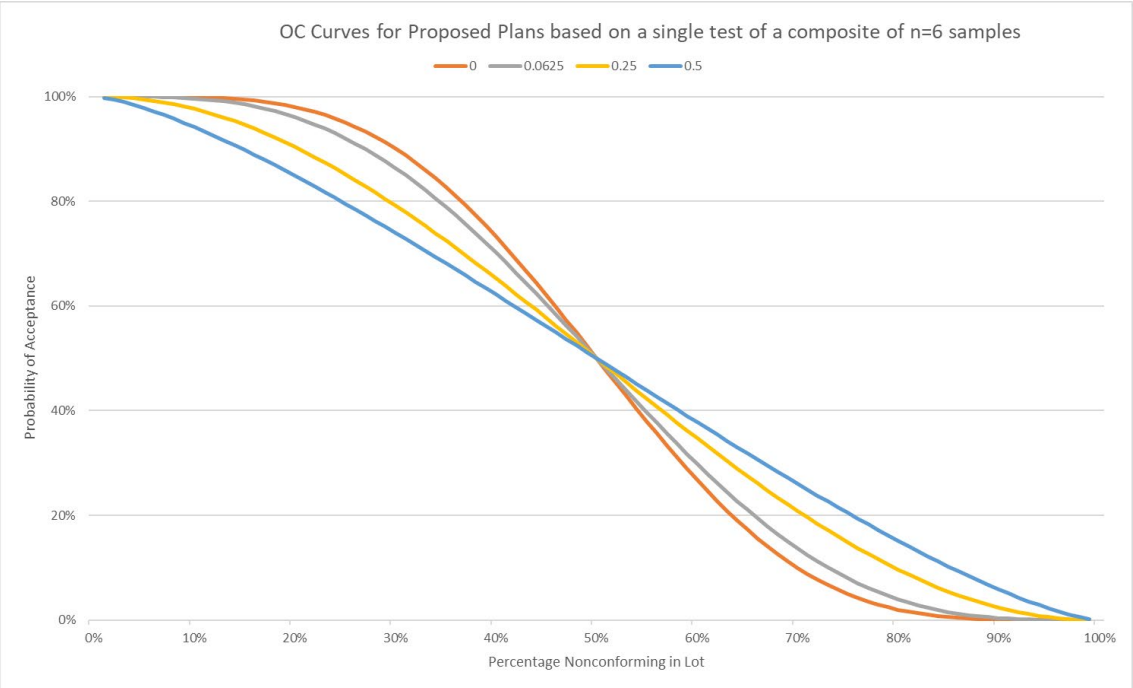
3.4.2.2 Attributes Plans

The probability that a single result will meet the upper limit is given by:

$$p.acc = NORMSDIST\big(NORMSINV(1 - NC) * sigma/SQRT(sigma^2/n + u^2)\big)$$

where NC is the percentage nonconforming in a lot.

Figure 30 OC curves - ad hoc attributes plans



Conclusion

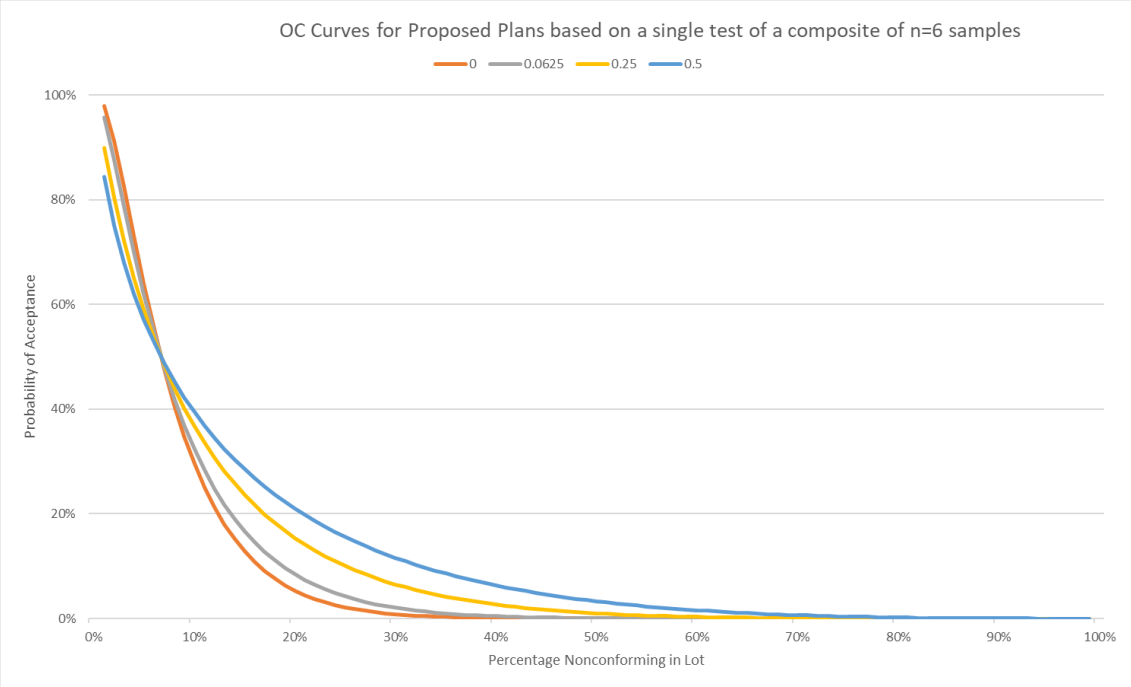
This plan also appears ineffective for assessments against upper or lower limits; this is not surprising since a composite sample represents the average level in a lot. Note that a single sample is also a representation of the average level of a lot.

Refer to section 4.1.2 for a discussion about (n=1, c=0) sampling plans.

3.4.2.3 Variables Plans

$$p.acc = NORMSDIST((NORMSINV(1 - NC) - k) * sigma/SQRT(sigma\text{^}2/n + u\text{^}2)))$$

Figure 31 OC curves - ad hoc variables plans



Conclusion

By using variables plans one can better control the risks of noncompliance by varying the value of k, the acceptability constant; the image shows the OC curves for k=1.5.

However, use by consumers of large k values for simplification or to reduce costs of testing does not seem to be a fair practice, considering that CXG50 is intended to apply mostly to commodity characteristics such as composition of 'commodity defects' and should serve to facilitate trade.

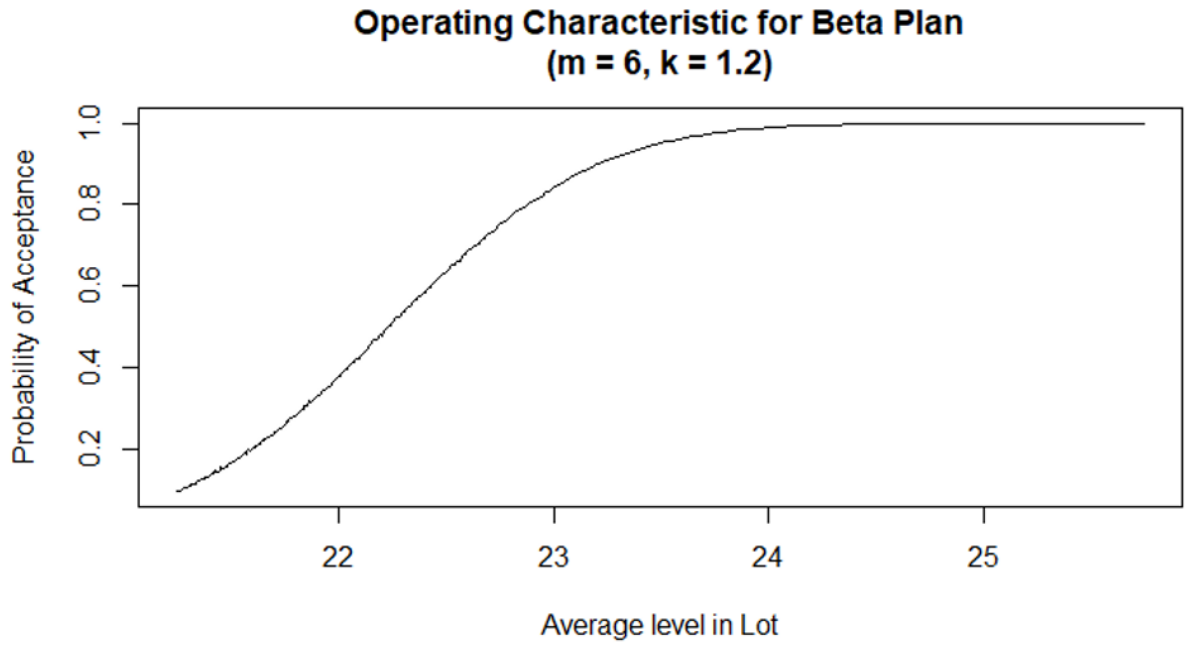
3.4.2.4 Beta distribution plans

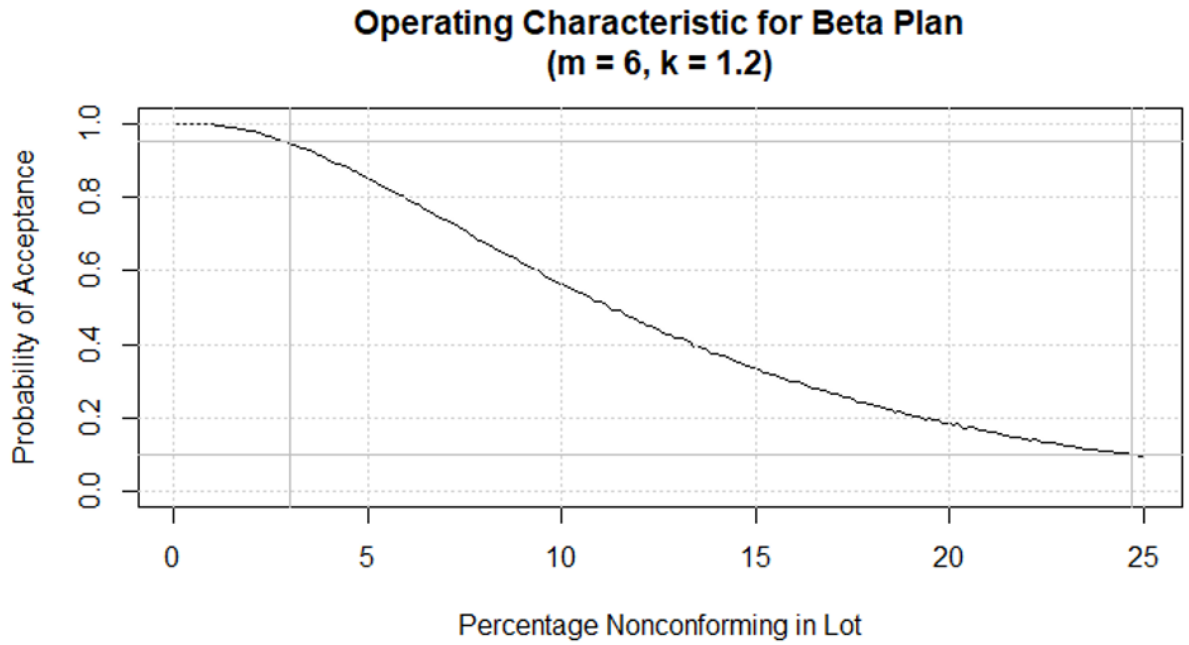
Use of a composite sample does not allow the use of attributes plans, and conventional variables plans won't be very useful unless one is assessing compliance against an average level (and maybe not even then).

The only possible classical solution seems to be the plans based on the beta distribution that requires the characteristic to be a compositional proportion and that measurement uncertainty is negligible.

These examples are based on a value of the theta parameter of θ = 500 and a minimum limit of L=20, (some previous work found that theta values for fat, protein and moisture in milk powders were between 700-3000).

Figure 32 OC curves for ad hoc beta plan





PART TWO

Background on sampling plans

Statistical appendix

4 Background to Acceptance Sampling Plans

4.1 Attributes plans

Two class attributes plans are based on the binomial distribution; for the plan (n, c) the probability of acceptance is given by:

$$Prob\ acceptance = \sum_{k=0}^{c} \binom{n}{k} p^k (1-p)^{n-k}$$

where p is the percentage nonconforming in the lot. This formula can be used to calculate the probability of acceptance for any level nonconforming p, to construct the operating characteristic.

This expression can be conveniently evaluated using the Excel function BINOM.DIST().

Example, the probability of accepting a lot in which p= 10% of the items are nonconforming, using the sampling plan (n=10, c=1) is given by.

BINOM.DIST(1,10,0.1,TRUE) = 0.736

or by the formula:

$$p_{acc} = \binom{10}{0} 0.9^{10} + \binom{10}{1} 0.9^9 \times 0.1 = 0.9^{10} + 10 \times 0.9^9 \times 0.1 = 0.736$$

However, if the level nonconforming varies between lots, this OC curve will not properly reflect the longer-term probability of acceptance with this plan; one solution due to Calvin [14] is to describe the variation in the level conforming by a beta distribution, in which case the long-term probability of acceptance will be given by a Polya distribution.

In general, if both the consumer's and producer's risks are specified in the design of the plan, as might be appropriate for non-food safety characteristics such as commodity defects, it is unlikely that the acceptance numbers, the c values, will be zero. It should be noted that rather large sample sizes (and large acceptance numbers) might be needed for plans where the operating ratio (CRQ/PRQ) is small.

Section 2.4.2 contains an Excel file that allows the calculation of 95% confidence intervals for the percentage nonconforming or the number of defects in a lot when nonconforming items have been found in the sample.

The lower 95% confidence limit (LCL) for the proportion of nonconforming items in the lot can be calculated using the Excel formula:

LCL = BETAINV(0.025, x, n-x+1),

and the upper 95% confidence limit by:

UCL = BETAINV(0.975, x+1, n-x)

where x is the number of nonconforming items observed in the sample and n is the total sample size.

Similarly, the lower 95% confidence limit (LCL) for the number of defects in the lot can be calculated using:

LCL = 2*GAMMA.INV(0.025, x, 0.5),

and the upper 95% confidence interval by:

UCL = 2*GAMMA.INV(0.975,x+1,0.5)

4.2 Variables plans

In the case of variables plans, once PR, CR, PRQ and CRQ have been specified, then the sample size n and the acceptance constant k can be calculated as follows:

$$k = \frac{z_{1-PR} \cdot z_{1-CRQ} - z_{1-PRQ} \cdot z_{CR}}{z_{1-PR} - z_{CR}}$$

where, for $0 < p < 1$, z_p denotes the one-sided quantile of a standard normal distribution, i.e.

$$\mathcal{P}(X \leq z_p) = p$$

for

$$X \sim \mathcal{N}(0,1).$$

In Excel, these quantiles can be calculated by means of the NORM.S.INV(p) function.

For the case that the lot standard deviation is known (σ method), the sample size can be determined as follows:

$$n = \left(\frac{z_{CR} - z_{1-PR}}{z_{1-CRQ} - z_{1-PRQ}} \right)^2$$

For the case that the lot standard deviation is not known (s method), the above expression for n must be multiplied by the factor $1 + \frac{k^2}{2}$.

The derivation of this concept is quite instructive and provided in Section 5.1.2.

4.2.1 Basis for calculations in App1

Firstly, when measurement uncertainty is negligible, the probabilities of acceptance for the variables sampling plans can be calculated using the formulas above, shown in terms of Excel functions as follows:

Known standard deviation 'sigma'

$$Prob.acc = NORMSDIST((NORMSINV(1 - theta) - k) * SQRT(n), TRUE)$$

Unknown standard deviation (sigma unknown, estimated from the inspection data):

$$Prob.acc = NORMDIST((NORMSINV(1 - theta) - k) * SQRT(n)/SQRT(1 + k * k/2))$$

where:

n is the number of samples,

k is the acceptability constant,

theta (θ) is the level nonconforming in the lot at which the probability of acceptance is to be calculated,

NORMSDIST() is the cumulative standard normal distribution function, and

NORMSINV() is the inverse of the standard normal distribution function.

There is no exact solution when the between laboratory standard deviation is non-negligible, so one must rely on an approximation. Wetherill [29] uses the following method based on a normal approximation for the negligible measurement uncertainty case.

$$p_{acc} = pr(\bar{x} + k \cdot s \leq U) = pr(\bar{x} + k \cdot s - \mu - k \cdot \sigma \leq U - \mu - k \cdot \sigma)$$

where k is the acceptability constant, U the upper specification limit and σ the lot standard deviation.

Making the substitution for $U - \mu = \sigma \cdot NORMSINV(1 - \theta)$, and standardising to a standard normal random variable Z~N(0,1) using the well-known normal approximations for the expected value and standard deviation (uncertainty) of a standard deviation:

$$E(s) = \sigma \; and \; var(s) = \frac{\sigma^2}{2(n-1)} \approx \frac{\sigma^2}{2n}$$

we get:

$$p_{acc} = pr\left(Z \leq \frac{NORMSINV(1-\theta) - k}{\sqrt{\frac{\sigma^2}{n} + \frac{k^2}{2n}}} \right) = NORMSDIST\left(\frac{NORMSINV(1-\theta) - k}{\sqrt{\frac{\sigma^2}{n} + \frac{k^2}{2n}}} \right)$$

This expression can be extended to allow for measurement uncertainty and any offsets used to allow for non-negligible between laboratory MU.

$$p_{acc} = pr\left(Z \leq \frac{NORMSINV(1-\theta) - k}{\sqrt{\sigma_b^2 + \frac{\sigma_r^2}{n} + \frac{\sigma^2}{n}}} \right) = NORMSDIST\left(\frac{NORMSINV(1-\theta) - k}{\sqrt{\sigma_b^2 + \frac{\sigma_r^2}{n} + \frac{\sigma^2}{n}}} \right)$$

In this expression the uncertainty of the calculated mean value depends on the repeatability standard deviation σ_r and the between laboratory standard deviation σ_b.

If an offset ($offset = q.\sigma_b$) is applied to allow for between laboratory measurement uncertainty, the probability of acceptance becomes.

$$p_{acc} = pr\left(Z \leq \frac{NORMSINV(1-\theta) - k - q.\sigma_b}{\sqrt{\sigma_b^2 + \frac{\sigma_r^2}{n} + \frac{\sigma^2}{n}}}\right) = NORMSDIST\left(\frac{NORMSINV(1-\theta) - k - q \cdot \sigma_b}{\sqrt{\sigma_b^2 + \frac{\sigma_r^2}{n} + \frac{\sigma^2}{n}}}\right)$$

where q is the multiplier of between laboratory standard deviation in the offset. Allowance for method biases can be made in the same way, subtracting the method bias from the numerator in the equation.

4.3 Calculating measurement uncertainty from precision estimates

The calculation of the probability of acceptance and the form of the acceptability criterion must take account of how the sampling and analytical components of measurement uncertainty are affected by any compositing, or by averaging of results performed as part of the overall sampling, subsampling, sample preparation and analytical procedures for the plan.

The following examples show the basic principles, where:

σ_r is the repeatability standard deviation

σ_L is the standard deviation representing the laboratory bias

σ_R is the reproducibility standard deviation

u is the standard measurement uncertainty

σ is the lot standard deviation

We distinguish the following 5 cases:

1. A single sample (increment) taken from a lot.

   $$u = \sigma_R = \sqrt{\sigma_L^2 + \sigma_r^2}$$

   This is the analytical component of measurement uncertainty.

2. A single taken from a lot, interpreting the result as the average level of the lot.

   $$u = \sqrt{\sigma^2 + \sigma_L^2 + \sigma_r^2}$$

3. n samples taken from lot, and tested, and the results averaged to provide an estimate of the average level

   $$u = \sqrt{\frac{\sigma^2}{n} + \sigma_L^2 + \frac{\sigma_r^2}{n}}$$

4. A composite of n subsamples is tested once to provide an estimate of the average level.

   $$u = \sqrt{\frac{\sigma^2}{n} + \sigma_L^2 + \sigma_r^2}$$

5. n samples taken from the lot, each is tested m times, and results averaged to estimate the average level

   $$u = \sqrt{\frac{\sigma^2}{nm} + \sigma_L^2 + \frac{\sigma_r^2}{nm}}$$

4.4 Combined attributes-variables plans

It is possible to modify the acceptance criterion for variables plans by including an additional requirement on the individual analytical results, typically that none of the results should exceed the specification limit. This leads to a combined attributes-variables plan.

This additional requirement will reduce the probability of acceptance, the decrease is obviously greater at higher levels nonconforming.

Refer also to CXG 50-2004 section 5.2.9 Fractional Nonconformance plans that are another type of combined attribute-variables plans.

4.5 Multi-stage plans

In multi-stage plans the inspection is carried out in several stages, most commonly two-stage plans are used. At each stage, a specified number of samples is taken and tested, although practically, a larger number of samples may be taken at the first stage in case they need to be tested at Stage 2:

- if the results meet the acceptance criterion for that stage, the lot is accepted without any further inspections needed.
- If the results meet the rejection criterion for that stage, the lot is rejected.
- If neither criterion is met, sampling continues to the next stage [if there is one].

The following example shows how a double attributes sampling plan operates. This example is based on a producer's risk of 5% at a quality level of 1% nonconforming and a consumer's risk with a 10% at a quality level of 5% nonconforming.

Stage 1:

n1 = 88 samples are taken at random from a lot.

- If at most one nonconforming item was found, then accept the lot.
- If four or more nonconforming items were found, then reject the lot.
- If two or three nonconforming items were found, proceed to Stage 2.

Stage 2:

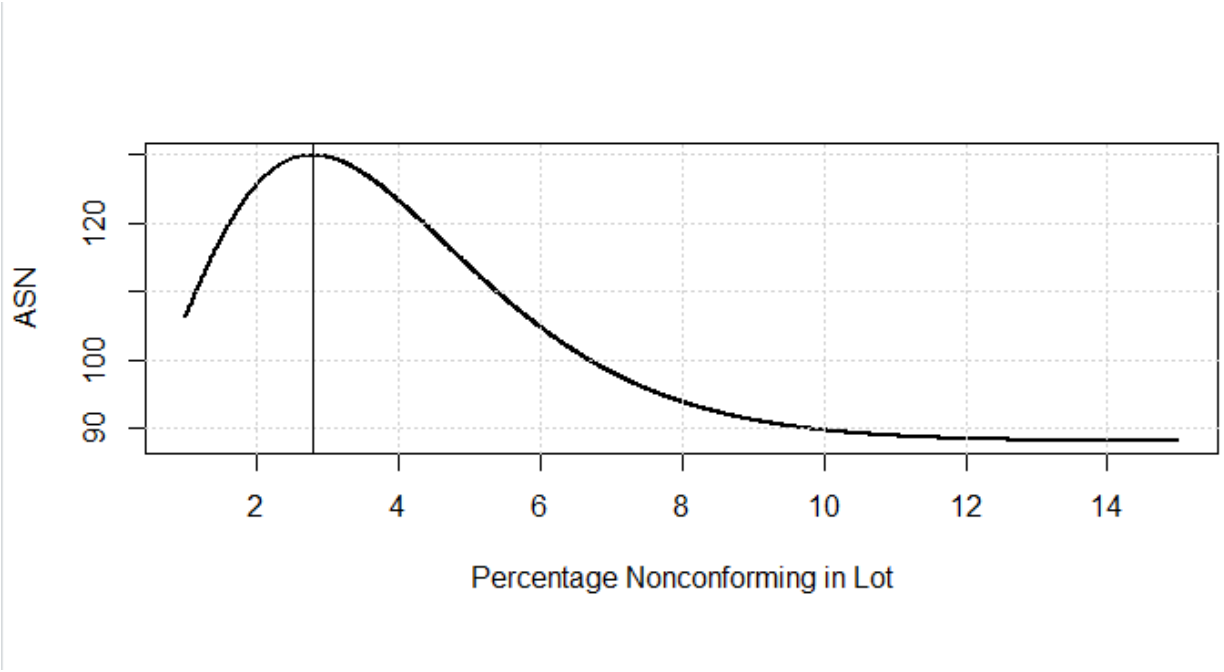
n2 = 88 additional samples are taken at random from a lot.

- If at most four nonconforming items were found in both stages, then accept the lot.
- If five or more nonconforming items were found in both stages, then reject the lot.

The main advantage of multi-stage plans is the reduction in the overall average sample size relative to the sample size for a single stage plan for the same control of producer's and consumer's risks; lots of very good quality lots are accepted, and lots of very poor quality are rejected, at the first stage. However, a disadvantage of multi-stage plans is the increased administrative and other costs and the possible delay making a final decision on the disposition of marginal lots.

The maximum Average Sample Number (ASN) for the double sampling plan is n1 + n2 = 130 approximately, at a quality level of about 2.8% nonconforming, but the ASN is considerably less at other levels nonconforming.

Figure 33: Two stage plan – expected average sample number (ASN) by level nonconforming



The corresponding single sample plan is (n=132, c=3).

4.6 Lots consisting of bulk materials

This section provides information on the design of plans for bulk materials, particularly relating to plans to assess compliance of the average level to a maximum or minimum limit that are often used for chemical contaminants. In particular, this section provides:

- The scope, some understanding, few basic properties of sampling plans for bulk materials, and 'motivation' for their use.
- Review of ISO 10725, Acceptance sampling plans and procedures for the inspection of bulk material, for assessment of the average level.
- Acceptance sampling for aflatoxins, in particular, the plans described in Whitaker's work, including explanation of tables from the *General standard for contaminants and toxins in food and feed* (CXS193-1995).

Detailed guidance is not provided on the following topics because of the statistical complexity involved; it is recommended that users seek assistance from a statistician:

- Characterizing the heterogeneity in bulk sampling, partitioning total heterogeneity in various components.
- The design of sampling plans for bulk materials to assess compliance against a minimum or maximum limits.

Note: plans for bulk materials are generally one-off i.e. applicable to a specific situation or a limited range of situations, so are not necessarily transferable to other matrices or characteristics.

Bicking (1970) defines the following process for the design of sampling plans for bulk materials:

1. State the problem for which an estimate of the average value is required.
2. Collect information on the relevant properties of the material (averages and components of variance of the properties)
3. Identify the components of variation in the overall sampling and testing process that might be relevant to the intended sampling plan options.
4. Estimate these components using a suitable statistical design (often 'hierarchical' designs are used)
5. Consider various approaches, taking account of cost, precision and difficulties.
6. Evaluate these plans in terms of the cost of sampling and testing, delay, supervisory time and convenience.
7. Calculate the standard deviations associated with the estimates of the average levels for these plans and their uncertainty (degrees of freedom).
8. Provisionally, select a plan from one of these approaches.
9. Reconsider the preceding steps.

The acceptance criterion will be of the form: $\bar{x} + k \cdot S \leq USL$ for upper specification limit USL for the average level, where *S* is the standard error (standard deviation) of the estimate $\bar{x}$ of the mean level and k is the multiplier[5] of the standard error in the acceptance criterion. Note that this multiplier is different from the acceptability constant used with variables plans used to control the percentage nonconforming.

If the cost associated with the initial sampling step was low the plan could be economized by taking more increments to improve the precision of the estimate of the average level.

4.6.1 Example: Variables Plans for homogeneous lots—negligible MU

As an alternative to the sampling plan discussed in section 4.2.1, the contents of the cans in the lot could be considered as a bulk material so that the assessment relates to the contents of the lot as a whole rather than to compliance at the can level. This approach would also allow plans for the beta distribution to be used, with the possible benefit of being able to carry out an inspection based on a single test of a composite sample, if the characteristic inspected was a compositional proportion.

[5] The multiplier is based on the Student's T-distribution for which the number of degrees of freedom has to be determined using a statistical procedure. See Schilling for an example and the CXG 54 information document for more information, but the details of this procedure are outside the scope of these guidelines.

If both producer's and consumer's risks are specified in the design of the sampling plan, the design process is the same as in section 4.2.1, noting that the sample size does not depend on the lot size if both risks are specified.

To avoid repetition, the full details of the plan in section 4.2.1 are not repeated here, but in summary that plan was based on the following assumptions:

The CRQ was chosen as 10% and the PRQ as 2.5%, with the CR and PR at 10% and 5% respectively. This means that the plan will have:

- A 10% chance of accepting a lot in which 10% of the product is nonconforming.
- A 5% chance of rejecting a lot in which 2.5% of the product is nonconforming.

The lot standard deviation was assumed to be $\sigma = 0.3$ and the measurement uncertainty was considered negligible, leading to the plan (n=43, k=1.59), i.e. 43 samples need to be taken from the lot and tested, with the lot is accepted with respect to milkfat provided the average and the standard deviation of the results meet the acceptance criterion:

$$\bar{x} - 1.59 \times s \geq 26$$

where:

- $\bar{x}$ is the average of the 43 individual results and 's' their standard deviation,

4.6.2 Example: Variables plan with non-negligible MU with no laboratory bias

Refer to section 4.2.2 – the process for the design of the sampling plan is the same.

4.6.3 Example: Variables plan with non-negligible MU with laboratory bias

Refer to section 4.2.3 – the process for the design of the sampling plan is the same as described in that section.

4.6.4 ISO 10725

This standard follows the work by Schilling & Neubauer and is discussed in their book [3], available on-line [5].

ISO10725 describes procedures for the design of sampling plans for the assessment of the average levels of lots, based on a three-component model:

- A number of increments are taken from the lot and combined to form composite samples.
- Test portions are taken from each of the well-mixed composite samples.
- Each test portion is tested a number of times.

As well as the variation of each component, the standard also allows for the actual (or relative) costs of each step to be taken into account to obtain cost optimal plans for specified levels of producer's and consumer's risks.

It is assumed that the standard deviations and the costs of each of the steps are known but the standard contains procedures to deal with situations where the costs or the standard deviations are not known.

4.6.5 Aflatoxin sampling plans

Introduction

The sampling plans for mycotoxins derived by Whitaker et al. are special cases of plans for bulk materials. Whitaker used 46 years of laboratory data, including some from contaminated lots, to derive Horwitz-type equations for the sampling, subsampling and analytical components of the total variation.

This method cannot be applied for the design of plans for new matrices or for new contaminants for which limited, or possibly unsuitable, historical data is available. In this case the classical approach described in Schilling, that underpins the ISO10725 standard, would be applied. A first step is to quantify the components of variation relevant to the intended sampling procedure using a suitable experimental design.

However, there are potential problems in that not every lot will be contaminated, and contamination might not be found in those lots that are actually contaminated, so that a considerable number of lots might be required for this exercise. Bayesian approaches might provide a way forward.

Following that, a sampling plan can be developed in terms of:

- the number of segments sampled
- the number of samples taken from each segment

- compositing and subsequent subsampling of those samples
- compositing of the subsamples
- the number of laboratory samples taken for testing
- analytical measurement uncertainty

The usual approach involves experimentation with the number of segments sampled, number of 'samples' taken at each stage and the number of results that are produced, the results from which are averaged. These numbers might also be chosen taking account of the cost involved in each operation. The statistical objective of the design process is the find the acceptability constant K in the acceptance criterion:

$$\bar{X} + t \cdot S \leq U$$

where:

U is the average test result

$\bar{X}$ is the average test result, that will be an estimate of the overall average level in the lot

S is the standard deviation of the estimate of the average level, usually referred to as the 'standard error'

t is the multiplier of the standard error in the acceptance criterion, a percentage point on the "t-distribution", obtained using a statistical procedure by taking account of the uncertainties of the components of the sampling and measurement variation.

There may be other considerations in the design of plans, not taken into account in Whitaker's work, such as:

- Whether one can make use of an assumed distribution for the characteristic in the lot considering that the behaviour of heterogenous materials cannot normally be explained in terms of a single standard deviation or distribution.
- Is it necessary to use discrete distributions to describe this behaviour given that the composite sampling will cause averaging?
- Does the use of cluster sampling need to be allowed for?

Other issues:

Designers of plans should consider what contamination scenarios they wish to detect, that is, the required probabilities of detection of detecting 'spikes' of contamination that contain certain levels of contamination and are of certain durations.

Compositing strategies should be developed to ensure that 'important' spikes of contamination are not averaged out to the extent that they cannot be found.

Aflatoxin sampling plans

The Horwitz type equations were derived for the three variance components (sample to sample, subsampling and the analytical components of variation) in terms of the average concentration of aflatoxin.

CXS 193 shows the breakdown of the total variation for aflatoxins in tree-nuts, for example, into components S_s^2, S_{sp}^2 and S_a^2, due to sampling, subsampling and testing respectively. It should be noted that provisions for aflatoxins are expressed in terms of the average levels in a lot; these plans employ large offsets between the limits and the levels at which the foods become unsafe to consume in order to provide consumer protection (refer CXG 50-2004 4.3.5 Plans for the average level in the 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	$S^2_s + S^2_{sp} + S^2_a$	$S^2_s + S^2_{sp} + S^2_a$	$S^2_s + S^2_{sp} + S^2_a$	$S^2_s + S^2_{sp} + S^2_a$

The sampling plan is defined in terms of laboratory sample size n_s, test portion size n_{ss} and the number of aliquots n_a, the number of analytical samples taken from each subsample. The information in this table can be used to calculate the uncertainty of the estimated average value obtained using the sampling procedure and

thereby the probability of acceptance. For example, the variance of the estimate of the average level for almonds is given by:

$$S^{2=}\frac{7730 \times 5.759}{n_s}C^{1.561} + \frac{100 \times 0.170}{n_{ss}}C^{1.646} + \frac{0.048}{n_a}C^2$$

This equation implies the following sampling and testing procedure:

1. n_s samples are taken from the lot under consideration.
2. A composite sample is formed.
3. A laboratory sample of size n_{ss} grams is taken from that well-mixed composite.
4. n_a aliquots are taken from that subsample for testing.

In the FAO Mycotoxin plans this procedure can be carried out on more than a single sample, but the results for the different samples are not averaged but compared with the limit separately.

This criterion differs from the usual acceptance criterion for the assessment of compliance of the average level for bulk materials in general that would be of the form:

$$\bar{X} + t \times S \leq USL$$

where S is the uncertainty of the average level, t is the multiplier of the standard deviation in the criterion and USL is the upper limit for the mean.

This is a further example of the use of offsets that, in this case, allow simplification of the acceptance criterion.

CXS 193-1995 describes the operational details of the sampling and testing procedure:

1. A 20kg sample taken (1000 [shelled] almonds per kg) from a lots or part lot (sublot), with a 25-tonne limitation on lot size. These samples should be formed from many smaller increments, each no less than 200g. CXS 193-1995 provides guidance on the number of increments, in terms of sample size.
2. The entire sample is ground to a uniform particle size and thoroughly mixed.
3. A test portion of no less than n_{ss} = 50g is taken from the composite sample
4. A number (n_a) of aliquots is taken for testing.
5. The results from these n_a tests are averaged. However, it appears that CXS 193-1995 assumes only single tests are performed (n_a =1) and that usually one or two different samples might be tested with the lot accepted provided no result exceeds the limit. This leads to different probabilities of acceptance, depending on the number of samples that are taken.

Example – Shelled Almonds for further processing

Suppose the average concentration of aflatoxins in the lot was C = 8 µg/kg and ns = 20000, 20 kg @ 1000 shelled nuts per kg, were taken as a sample, and this sample was ground, and well-mixed composite formed. If a subsample of 50g was taken and a single aliquot (n_a=1) tested, the standard deviation S representing the uncertainty of the average level would be:

$$S^{2=}\frac{7730 \times 5.759}{20 \times 1000}8^{1.561} + \frac{100 \times 0.170}{50}8^{1.646} + \frac{0.048}{1}8^2 = 70.67$$

Giving S = 8.41. The first component representing the sample-by-sample variation is much larger than the other two.

Comments

The web-based FAO Mycotoxin Tool [2] for the evaluation of sampling plans is provided at http://tools.fstools.org/mycotoxins/.

This tool allows for only a single component of measurement uncertainty; there is no allowance for bias when multiple tests are performed. The tool allows users to select whether “within lab” or “among lab” variation is used, with the among laboratory variation equal to twice the within laboratory figure. The tables below show the within laboratory variance.

The sampling component is included using an assumed distribution, most often the negative binomial, a discrete distribution to allow contamination at the individual particle (e.g. grain) or sample level to be modelled - due to the small percentages (typically less than 1%) of contamination and the extreme distribution of contamination within lots very large sample sizes are needed to estimate the distribution.

The decision rule for Almonds for further processing in CXS 193 is that the lot is accepted "if the aflatoxin result is less than 15µg/kg in both samples…", so that each individual result is classified as pass or fail with respect to the limit. However, as the analytical component is small relative to the sampling component, this does not seem to matter.

To calculate probabilities of acceptance (and the OC curve) we need to know the distribution of the sample-to-sample variation within a bulk lot. As above, Whitaker assumed, mostly, that the sample-to-sample variation follows a negative binomial distribution.

The negative binomial distribution is used in situations where the variation is more extreme than the binomial; it is defined in terms of an average value and a variance.

$$Average = \mu;\ Variance = S^2 = \mu + \frac{\mu^2}{k}$$

where ***k*** is the dispersion factor that allows for the extra variation.

To work out the theoretical probability of acceptance at a concentration C of aflatoxin Whitaker used the 'method of moments', equating the theoretical concentration C to the mean and the estimate of S^2 to the variance, i.e.

$$\mu = C\ and\ S^2 = C + \frac{C^2}{k}$$

The second equation is be solved to determine k and the probability of acceptance calculated. This process must be repeated for each value of C, as S^2 depends on C.

Components of Variance for Aflatoxin Sampling Plans

Study #	Mycotoxin	Commodity	References	Variance			Mycotoxin Test Procedure				Distribution Among Sample Test Results
				Sampling (S^2_s)	Sample Preparation (S^2_{sp})	Analytical (Within Lab) (S^2_a)	Laboratory Sample Size (ns)	Comminuted Test Portion Size (nss)	Number of Aliquots (na)	Concentration (C)	
1	Aflatoxin	Shelled Peanuts	1, 2, 3, 34	$(10.644/ns)9.19C^{1.335}$	$(275/nss)0.294C^{1.729}$	$(1/na)0.083C^{1.664}$	Number of shelled kernels (1,952ker/kg)	Mass (g) Dry Comminution USDA mill powder	Number of aliquots quantified by HPLC from Ref 34	ng/g (ppb) aflatoxin total	Negative Binomial
2	Aflatoxin	Cottonseed	4, 5, 6, 34	$(43,200/ns)6.776C^{1.344}$	$(200/nss)0.180C^{1.3308}$	$(1/na)0.086C^{1.667}$	Number of seed (Hull removed) (19,031ker/kg)	Mass (g) Dry Comminution USDA mill powder	Number of aliquots quantified by HPLC from Ref 34	ng/g (ppb) aflatoxin either total or B1	Negative Binomial
3	Aflatoxin	Harvested Inshell Peanuts (Farmer's Stock)	7, 8, 9	$(3713/ns)37.607C^{1.161}$	$(100/nss)2.887C^{1.401}$	$(1/na)0.083C^{1.664}$	Number of inshell pods (882pods/kg)	Mass (g) Dry Comminution VCM Paste	Number of aliquots quantified by HPLC from Ref 34	ng/g (ppb) aflatoxin total	Negative Binomial
4	Aflatoxin	Shelled Corn	10, 11, 12	$(3,390/ns)11.36C^{0.98}$	$(50/nss)1.254C^{1.27}$	$(1/na)0.143C^{1.16}$	Number of shelled kernels (3,000ker/kg)	Mass (g) Dry Comminution Romer Powder	Number of aliquots quantified by HPLC	ng/g (ppb) aflatoxin total	Compound Gamma Used Negative Binomial
5	Aflatoxin	Shelled Almonds	13, 14, 15	$(7,730/ns)5.759C^{1.581}$	$(100/nss)0.170C^{1.646}$	$(1/na)0.0041C^{1.986}$	Number of shelled kernels (773ker/kg)	Mass (g) Dry Comminution VCM Paste	Number of aliquots quantified by HPLC	ng/g (ppb) aflatoxin either total or B1	Negative Binomial
6	Aflatoxin	Inshell Almonds	13, 14, 15	$(7,730/ns)5.759C^{1.581}$	$(100/nss)0.170C^{1.646}$	$(1/na)0.0041C^{1.986}$	Number of Inshell Nuts (309nuts/kg) Shell/ker Ratio = 60/40	Mass (g) Dry Comminution VCM Paste	Number of aliquots quantified by HPLC	ng/g (ppb) aflatoxin either total or B1	Negative Binomial
7	Aflatoxin	Shelled Hazelnuts	15, 16, 17	$(10,000/ns)4.291C^{1.609}$	$(50/nss)0.021C^{1.645}$	$(1/na)0.0028C^{1.990}$	Number of shelled kernels (1,000ker/kg)	Mass (g) Dry Comminution VCM Paste	Number of aliquots quantified by HPLC	ng/g (ppb) aflatoxin either total or B1	Negative Binomial
8	Aflatoxin	Inshell Hazelnuts	15, 16, 17	$(10,000/ns)4.291C^{1.609}$	$(50/nss)0.021C^{1.645}$	$(1/na)0.0028C^{1.990}$	Number of Inshell nuts (500Nuts/kg) Shell/Ker Ratio = 50/50	Mass (g) Dry Comminution VCM Paste	Number of aliquots quantified by HPLC	ng/g (ppb) aflatoxin either total or B1	Negative Binomial
9	Aflatoxin	Shelled Pistachios	15	$(8,000/ns)7.913C^{1.475}$	$(25/nss)2.334C^{1.622}$	$(1/na)0.0368C^{1.598}$	Number of Shelled Kernels (1,600ker/kg)	Mass (g) Dry Comminution VCM Paste	Number of aliquots quantified by HPLC	ng/g (ppb) aflatoxin either total or B1	Negative Binomial
10	Aflatoxin	Inshell Pistachios	15	$(8,000/ns)7.913C^{1.475}$	$(25/nss)2.334C^{1.622}$	$(1/na)0.0368C^{1.598}$	Number of Inshell Nuts (800nuts/kg) Shell/Ker Ratio = 50/50	Mass (g) Dry Comminution VCM Paste	Number of aliquots quantified by HPLC	ng/g (ppb) aflatoxin either total or B1	Negative Binomial

Study #	Mycotoxin	Commodity	References	Variance			Mycotoxin Test Procedure				Distribution Among Sample Test Results
				Sampling (S^2_s)	Sample Preparation (S^2_{sp})	Analytical (Within Lab) (S^2_a)	Laboratory Sample Size (ns)	Comminuted Test Portion Size (nss)	Number of Aliquots (na)	Concentration (C)	
11	Aflatoxin	Shelled Brazil Nuts	15	$(1{,}850/ns)4.862C^{1.889}$	$(50/nss)0.0306C^{0.632}$	$(1/na)0.0164C^{1.117}$	Number of Shelled Kernels (185ker/kg)	Mass (g) Slurry (Water/Ker 1/1) Comminution	Number of aliquots quantified by HPLC	ng/g (ppb) aflatoxin either total or B1	Negative Binomial
12	Aflatoxin	Inshelled Brazil Nuts	15	$(1{,}850/ns)4.862C^{1.889}$	$(50/nss)0.0306C^{0.632}$	$(1/na)0.0164C^{1.117}$	Number of Inshelled Nuts (93Nuts/kg) Shell/Ker Ratio=50/50	Mass (g) Slurry (Water/Ker 1/1) Comminution	Number of aliquots quantified by HPLC	ng/g (ppb) aflatoxin either total or B1	Negative Binomial
13	Aflatoxin	In Field Ear Corn	18	$(600/ns)8.919C^{2.230}$	$(50/nss)1.254C^{1.27}$	$(1/na)0.143C^{1.16}$	Number of shelled kernels per ear 200 g ker/ear (3,000ker/kg)	Mass (g) Dry Comminution Romer Powder	Number of aliquots quantified by HPLC	ng/g (ppb) aflatoxin B1	Negative Binomial
14	Aflatoxin	In Field Farmer's Stock Peanuts	19	$(116/ns)17.056C^{1.6686}$	$(100/nss)2.887C^{1.401}$	$(1/na)0.083C^{1.654}$	Number of inshell pods (882pods/kg)	Mass (g) Dry Comminution VCM Paste	Number of aliquots quantified by HPLC from Ref 34	ng/g (ppb) aflatoxin total	Negative Binomial
15	Aflatoxin	Powdered Ginger in Capsules	20	$(5/ns)0.138C^{1.0}$	No Test Portion, Entire Sample Extracted	$(1/na)0.0178C^{1.70}$	5 g Laboratory Sample is also the 5 g Test Portion	No Test Portion, Entire Sample Extracted	Number of aliquots quantified by HPLC	ng/g (ppb) aflatoxin total	Normal
16	Aflatoxin	Powdered Ginger in 1-Lb Bags	21	$(5/ns)4.218C^{1.0}$	No Test Portion, Entire Sample Extracted	$(1/na)0.00349C^{1.70}$	5 g Laboratory Sample is also the 5 g Test Portion	No Test Portion, Entire Sample Extracted	Number of aliquots quantified by HPLC	ng/g (ppb) aflatoxin total	Normal
17	Aflatoxin	Dried Figs	Not Published	$(590/ns)2.219C^{1.433}$	$(55/nss)0.012C^{1.465}$	$(1/na)0.006C^{1.368}$	Number of dried Figs (59 Figs/kg)	Mass (g) Slurry (Water/Ker 1/1) Comminution	Number of aliquots quantified by HPLC	ng/g (ppb) aflatoxin total	Negative Binomial
18	Fumonisin	Shelled Corn	22, 23, 24	$(3{,}390/ns)0.033C^{1.75}$	$(25/nss)0.011C^{1.59}$	$(1/na)0.014C^{1.44}$	Number of shelled kernels (3,000ker/kg)	Mass (g) Dry Comminution Romer Powder	Number of aliquots quantified by HPLC	ug/g (ppm) Fumonisin either B1, B2, B3 or total	Compound Gamma Used Lognormal
19	Deoxynivalenol (DON)	Shelled Corn	25	$(3{,}000/ns)0.202C^{1.923}$	$(50/nss)0.0193C^{1.140}$	$(1/na)0.0036C^{1.507}$	Number of shelled corn kernels (3,000ker/kg)	Mass (g) Dry Comminution Romer - 25 g	Number of aliquots quantified by Romer - Malone HPLC	ug/g (ppm) DON	Lognormal (not published)
20	Deoxynivalenol (DON)	Wheat	26	$(13{,}620/ns)0.026C^{0.833}$	$(25/nss)0.066C^{0.833}$	$(1/na)0.026C^{0.833}$	Number of raw wheat kernels (30,000ker/kg)	Mass (g) Dry Comminution Romer 25 g	Number of aliquots quantified by Romer FluoroQuant	ug/g (ppm) DON	Lognormal (not published)

Study #	Mycotoxin	Commodity	References	Variance			Mycotoxin Test Procedure				Distribution Among Sample Test Results
				Sampling (S^2_s)	Sample Preparation (S^2_{sp})	Analytical (Within Lab) (S^2_a)	Laboratory Sample Size (ns)	Comminuted Test Portion Size (nss)	Number of Aliquots (na)	Concentration (C)	
21	Deoxynivalenol (DON)	Barley	27	$(77{,}000/ns)0.0122C^{0.947}$	$(50/nss)0.003C^{1.956}$	$(1/na)0.0108C^{1.055}$	Number of raw barley kernels (30,800ker/kg)	Mass (g) Dry Comminution Romer 50 g	Number of aliquots quantified by Romer FluoroQuant	ug/g (ppm) DON	Lognormal (not published)
22	Ochratoxin A (OTA)	Green Coffee Beans	28, 29, 30	$(1{,}500/ns)1.350C^{1.090}$	$(25/nss)0.272C^{1.646}$	$(1/na)0.008C^{1.605}$	Number of beans (1,500ker/kg)	Mass (g) Dry Comminution VCM Paste	Number of aliquots quantified by HPLC	ng/g (ppb) OTA total	Lognormal
23	Ochratoxin A (OTA)	Powdered Ginger in Capsules	20	$(5/ns)0.108C^{1.0}$	No Test Portion, Entire Sample Extracted	$(1/na)0.00654C^{1.70}$	5 g Laboratory Sample is also the 5 g Test Portion	No Test Portion, Entire Sample Extracted	Number of aliquots quantified by HPLC	ng/g (ppb) OTA total	Normal
24	Ochratoxin A (OTA)	Powdered Ginger in 1-Lb Bags	21	$(5/ns)1.336C^{1.0}$	No Test Portion, Entire Sample Extracted	$(1/na)0.00146C^{1.70}$	5 g Laboratory Sample is also the 5 g Test Portion	No Test Portion, Entire Sample Extracted	Number of aliquots quantified by HPLC	ng/g (ppb) OTA total	Normal
25	Ochratoxin A (OTA)	Oats	Not Published	$(55{,}796/ns)1.440C^{1.278}$	$(100/nss)0.0074C^{1.638}$	$(1/na)0.0103C^{1.58}$	Number of raw oat kernels (27,898ker/kg)	Mass (g) Dry Comminution Retsch SR300 #20 Screen	Number of aliquots quantified by HPLC	ng/g (ppb) OTA total	Negative Binomial
26	Ochratoxin A (OTA)	Wheat	Not Published	$(60{,}180/ns)1.557C^{1.132}$	$(5/nss)0.207C^{1.152}$	$(1/na)0.0204C^{1.866}$	Number of raw wheat kernels (30,090ker/kg)	Mass (g) Dry Comminution Retsch SR300 #20 Screen	Number of aliquots quantified by HPLC	ng/g (ppb) OTA total	Negative Binomial
27	FAPAS among lab variability		31			$(1/na)0.0484C^{2.000}$					
28	Horwitz among lab variability (ppb)		32,33			$(1/na)0.2048C^{1.70}$					
29	Whitaker, Horwitz, Analytical Variances - TLC, Immuno, HPLC		34			Among Lab = 2*Within Lab					

Study 3 the sampling variance was calculated by subtracting analytical and sample prep variances from total variances for each of the three (2.26, 4.21, and 6.91 kg) sample sizes.

Studies 13 and 14 measured only total variance. Used sample prep and analytical variances from studies 4 and 3, respectively.

Study 28 analytical variance was determined for various methods, mycotoxins, and commodities using data base from Horwitz Ref 32

4.6.6 General plans for assessment against minimum or maximum levels

One approach, more suited to food safety than commercial characteristics on the grounds of fairness, is to use offsets and assess compliance of lots against the average level. This has the considerable advantage of simplicity.

However, these plans are also important in a commercial context where one might, for example, wish to provide assurance about the average level of protein in a lot of grain that is to be further processed, for example to make flour.

In general, however, the design of sampling plans for bulk materials to assess compliance against a minimum or maximum limit is difficult statistically and no information is included in this information document.

4.7 Plans for microbiological assessment

Plans used for the assessment of lots for microbiological characteristics, often referred to as *Microbiological Criteria*, frequently employ 2-class attributes plans that require n=5 samples to be taken. These plans are suitable only for characteristics where the measurements are counts and there are adequate offsets between the limits used in these plans and the levels at which foods are considered to be unsafe.

If the offsets are not adequate there could be a higher rate of acceptance of contaminated product. Testing of pathogens is usually carried out using detection tests that produce presence or absence outcomes; in this case there are no offsets between the limits (zero) and the levels at which foods become unsafe to consume. For this reason, the use of (n=5, c=0) plans for pathogens is inadvisable; this is also the reason why sampling plans for pathogenic characteristics require much larger sample numbers and a greater total amount of sample is tested. Use of a larger numbers of samples also provides some safeguard against potentially inhomogeneous contamination within lots. Some examples of microbiological criteria are given in the *Code of hygienic practice for powdered formulae for infants and young children* (CXC 66-2008) that contains the following microbiological criteria (see Codex definition at the end of the section) along with some points on the Operating Characteristic:

Microorganism	n	c	m	Class Plan
Cronobacter sp.	30	0	0/10g	2
Salmonella	60	0	0/25g	2

Points on the operating characteristic have been calculated by *Zweiterung et al*. [10] assuming a Poisson-lognormal distribution, being a Poisson distribution whose mean varies according to a lognormal distribution.

Cronobacter:

- At a mean concentration of 1 cfu/340g, the probability of detection is 95%, assuming a standard deviation [for the lognormal distribution] of sd = 0.8.
- At a mean concentration of 1 cfu/100g, the probability of detection is 99%, assuming a standard deviation of sd = 0.5.

Salmonella:

- At a mean concentration of 1 cfu/526g, the probability of detection is 95%, assuming a standard deviation of sd = 0.8.

4.7.1 3-class attribute plans

Refer CXG 50-2004 section 4.2.6

In these plans inspection results are classified into three classes, usually referred to as ‘good’, ‘marginal’ and ‘poor’ or ‘unacceptable’. They have an advantage, relative to two-class plans of providing better discrimination between good and poor quality i.e. 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’;
- Samples with results lying between the numbers m and M are classified as marginal.

Lots are accepted provided:

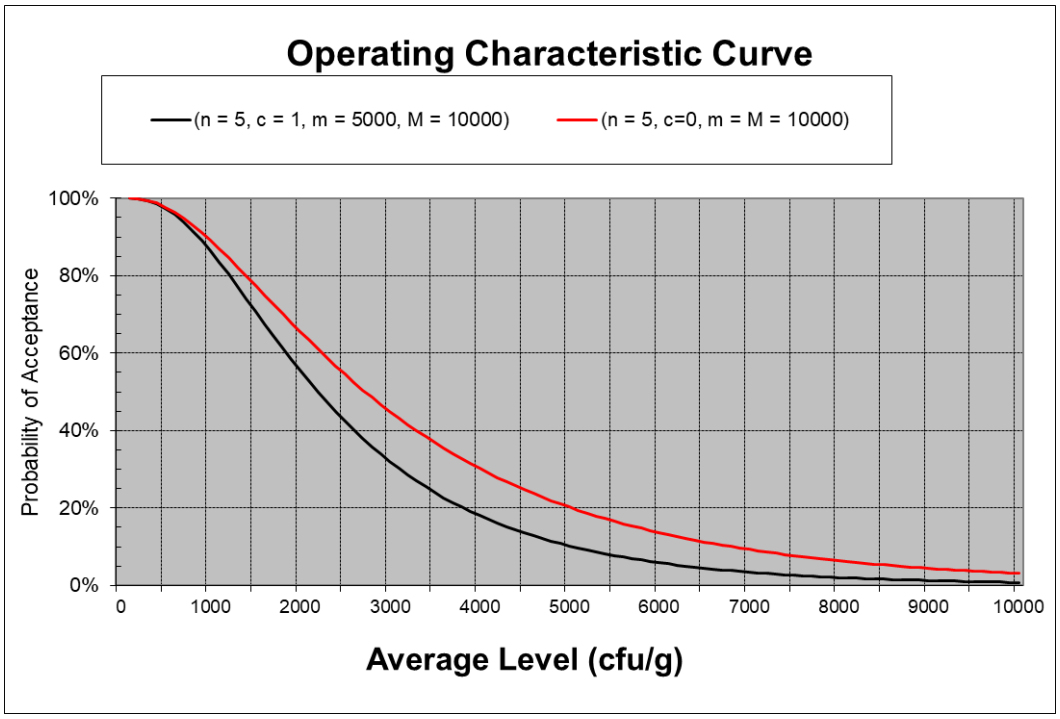
- None of the n samples is poor, having levels exceeding M
- At most c of the samples are marginal, with levels between m and M.

If m = 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, the lognormal distribution is commonly used for microbiological characteristics for counts occurring at higher levels, whereas the Poisson distribution is often used for counts at lower levels.

The following plot shows the operating characteristic curves for a two-class plan (n=5, c=0, m=10000) and a three-class plan (n=5, c=1, m=5000, M=10000); it shows that the three-class plan is more stringent despite allowing one result to be marginal.

Figure 34 OC curves - three class attributes plans



Although the plans mentioned in this section are used primarily in microbiological inspections, they are nevertheless useful in other applications such as those where acceptance is decided in terms of the total defects found in the sample, with the possibility that an item selected in the sample may contain more than one defect. One possible application of these plans is to the inspection of herbs and spices for insects or insect parts.

5 Statistical appendix

5.1 Background for the main (attributes & variables) sampling plans

5.1.1 Calculating acceptance probabilities – attributes plans

Attributes Plans are based on the binomial distribution (two-class plans) or the multinomial distribution, an extension of the binomial distribution, for three or more class plans.

The probability of acceptance for the two-class binomial model is given by:

$$prob\_acceptance = \sum_{k=0}^{c} \binom{n}{k} p^k (1-p)^{n-k}$$

where:

n is the sample size, the number or items or samples taken

c is the acceptance number, the maximum number of nonconforming items permitted for acceptance of the lot

p is the percentage nonconforming in the lot

For any given expression which depends on a variable k, the symbol

$$\sum_{k=value_1}^{value_n} \text{expression}(k)$$

means 'the sum of' the expression evaluated at

$$k = value_1,\ k = value_1 + 1,\ k = value_1 + 2,\ \ldots,\ k = value_n$$

For example

$$\sum_{k=1}^{5} k^2 = 1^2 + 2^2 + 3^2 + 4^2 + 5^2$$

The symbol $\binom{n}{k}$ is the binomial coefficient, i.e. it is the number of ways of choosing k items from a total of n items. For example, $\binom{5}{1} = 5$ since there are 5 ways of choosing one item from 5 items, viz. Aaaaa, aAaaa, aaAaa, aaaAa and aaaaA, where A represents the item selected.

The design of an attributes sampling plan involves finding the values of the number of samples n and the acceptance number c from the probabilities of acceptance at two specified points on the operating characteristic curve. Typically, these points are chosen as the producer's and the consumer's risk quality levels.

In the case p = PRQ, the probability of acceptance is equal to "one minus the producer's risk"

$$Prob\ acceptance = 1 - PR = \sum_{k=0}^{c} \binom{n}{k} p^k (1-p)^{n-k}$$

and when p = CRQ, the probability of acceptance CR is equal to the consumer's risk

$$Prob\ acceptance = CR = \sum_{k=0}^{c} \binom{n}{k} p^k (1-p)^{n-k}$$

These two equations are usually solved iteratively in a statistical package or using a computer program following the algorithm due to Hailey [15]:

1. Start by setting $n = 0$ and $c = 0$
2. If the probability of acceptance at the CRQ exceeds the specified maximum allowable consumer's risk CR, then increase n by one, and go back to step 2.
3. If the probability of rejection at the PRQ exceeds the specified maximum allowable producer's risk PR, then increase c by one, and go to step 2.

Note that because n and c are integers, and can only increase in steps of one, the actual producer's and consumer's risks in the final plan might not be exactly equal to the producer's and consumer's risks specified in the design of the plan.

Calculation of confidence intervals

Section 3.1.2 discussed ZAN plans and their use in applications such as inspections for foreign matter. The final part of that section described the calculation of confidence intervals for the percentage nonconforming or the number of defects in a lot when at least one nonconforming item or defect has been found in the samples examined.

For the binomial case that relates to the percentage of defective items in the lot overall, the lower and upper limits are calculated using the Excel formulas:

$$LCL = BETA.INV(0.025, c, n - c + 1)$$

and

$$UCL = BETA.INV(0.975, c + 1, n - c)$$

where n is the number of items or samples examined and c is the number of nonconforming items found among those n items.

For the Poisson case that relates to the percentage of defective items in the lot overall, the lower and upper limits are calculated using the Excel formulas:

$$LCL = 2 * GAMMA.INV(0.025, c, 0.5)$$

and

$$UCL = 2 * GAMMA.INV(0.975, c + 1, 0.5)$$

where n is the number of items or samples examined and c is the number of defects found during the inspection.

5.1.2 Derivation of formulas for variables plans

The formulas for k and n are derived as follows for the case of a known lot standard deviation σ and an upper specification limit U.

We use the notation z_p to denote the one-sided quantile of a standard normal distribution, i.e.

$$\mathcal{P}(X \leq z_p) = p$$

for

$$X \sim \mathcal{N}(0,1).$$

The acceptance limit A is defined as

$$A = U - k\sigma$$

We thus have

$$\begin{aligned} U - A &= k\sigma \\ &= \mu_{PRQ} + z_{1-PRQ} \cdot \sigma - \left(\mu_{PRQ} + z_{1-PR} \cdot \frac{\sigma}{\sqrt{n}}\right) && \text{Eq. 1} \\ &= \mu_{CRQ} + z_{1-CRQ} \cdot \sigma - \left(\mu_{CRQ} + z_{CR} \cdot \frac{\sigma}{\sqrt{n}}\right) && \text{Eq. 2} \end{aligned}$$

The following example and figure illustrate these two equations. Consider the case that we are asked to design a plan with

$$PRQ = 6.5\%$$

$$PR = 5\%$$

$$CRQ = 26\%$$

$$CR = 10\%$$

The corresponding standard normal quantiles are:

$$z_{1-PRQ} = 1.514$$

$$z_{1-PR} = 1.645$$

$$z_{1-CRQ} = 0.643$$

$$z_{CR} = -1.282$$

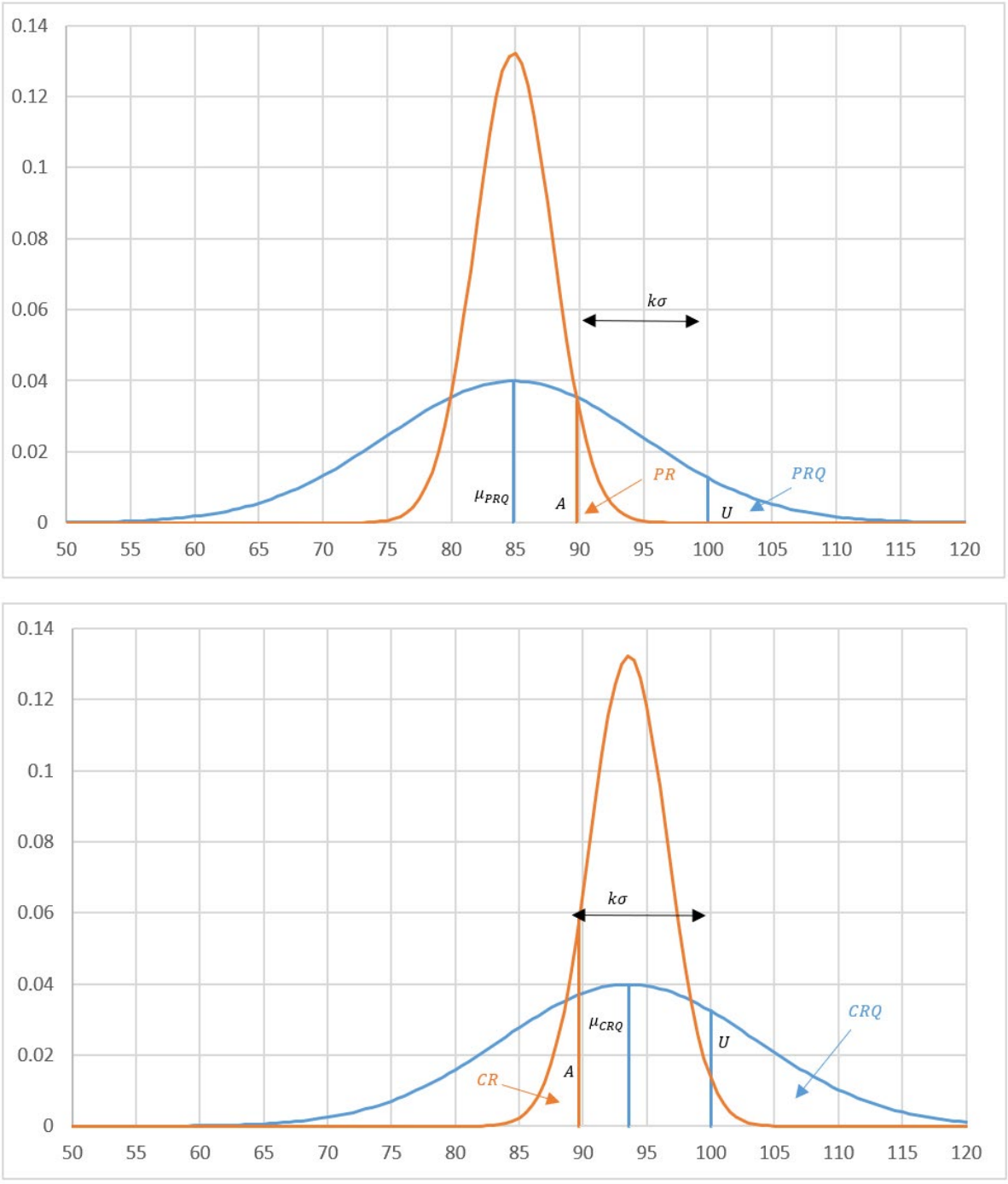
Applying the formulas for n and k (known σ), we obtain

$$n = 11.3$$

$$k = 1.025$$

This is illustrated in the following diagrams. We consider the situation that $U = 100$ (generic unit) and that the lot standard deviation is known with $\sigma = 10$. A lot with quality PRQ will have a mean value (across items) $\mu_{PRQ} \approx 85$. The sample size is $n = 11$. The acceptance limit (for the decision to accept or reject the lot) is calculated as $A = U - k\sigma \approx \mu_{PRQ} + z_{1-PR} \cdot \frac{\sigma}{\sqrt{n}} \approx 90$.

Figure 35: The blue curve represents the distribution of the property of interest in a lot with quality PRQ (top diagram) and in a lot with quality CRQ (bottom diagram) and lot standard deviation $\sigma = 10$. The red curve represents the statistical distribution of the arithmetic mean.



It follows from Equation 1 and Equation 2 that

$$\frac{1}{\sqrt{n}}(z_{CR} - z_{1-PR}) = z_{1-CRQ} - z_{1-PRQ}$$

And hence

$$\sqrt{n} = \frac{z_{1-PR} - z_{CR}}{z_{1-PRQ} - z_{1-CRQ}}$$

As far as k is concerned, it follows from Equation 1 and Equation 2 that

$$k = z_{1-PRQ} - \frac{z_{1-PR}}{\sqrt{n}}$$

$$k = z_{1-CRQ} - \frac{z_{CR}}{\sqrt{n}}$$

Hence, we have

$$\frac{k\sqrt{n}}{z_{1-PR}} = \frac{\sqrt{n} \cdot z_{1-PRQ}}{z_{1-PR}} - 1$$

and

$$\frac{k\sqrt{n}}{z_{CR}} = \frac{\sqrt{n} \cdot z_{1-CRQ}}{z_{CR}} - 1$$

It follows that

$$\frac{k\sqrt{n}}{z_{1-PR}} - \frac{k\sqrt{n}}{z_{CR}} = \frac{\sqrt{n} \cdot z_{1-PRQ}}{z_{1-PR}} - \frac{\sqrt{n} \cdot z_{1-CRQ}}{z_{CR}}$$

and thus

$$k \cdot \left(\frac{z_{CR} - z_{1-PR}}{z_{1-PR} \cdot z_{CR}}\right) = \frac{z_{1-PRQ} \cdot z_{CR} - z_{1-PR} \cdot z_{1-CRQ}}{z_{1-PR} \cdot z_{CR}}$$

From which we obtain

$$k = \frac{z_{1-PR} \cdot z_{1-CRQ} - z_{1-PRQ} \cdot z_{CR}}{z_{1-PR} - z_{CR}}$$

5.1.3 Within-item variability

In general, for a lot consisting of discrete items, there are two sources of variation: between-item variation and within-item variation. In “classical” acceptance sampling plans, there is a tacit assumption that within-item variation is negligible, and that one test result per item is thus sufficient. If non-negligible within-item variation is expected, then it may be necessary to modify the acceptance sampling plan. In particular, it may be necessary to correct the estimate of the lot standard deviation by “subtracting” the within-item component.

At low concentrations and for certain types of products (more specifically, when the presence or absence of the analyte is modelled via a discrete statistical distribution, as may be the case for sufficiently coarse-grained powders), within-item variation may be present even in the case of a “perfect” mixing process. This is due to an irreducible component of variation which remains even in the case of a perfectly homogenous item. This irreducible component is called the fundamental variability and is modelled via the Poisson distribution.

In the presence of fundamental variability, it may be necessary to apply specially developed models to distinguish between- from within-item variation. In particular, two different cases must be distinguished:

Case 1: the exact same quantity of analyte (corresponding to the property of interest, e.g. vitamin D in milk powder) is added separately to each item. Hence, there is no between-item variation, only within-item variation.

Case 2: the analyte is added to the mixing tank and then mixed with the pre-blend powder prior to filling the individual item recipients. In this case there is both between- and within-item variation.

Models for these two cases are discussed in Uhlig et al. (2025) [27].

5.2 Understanding ISO plans

The ISO standards apply a risk-based approach to the design of acceptance sampling plans. In inspection by attributes, it is the product AQL (PRQ) × sample size which informs the design of the plans. In inspection by variables, the plans indexed by AQL (PRQ) aim to achieve a producer's risk which depends on the lot size.

5.2.1 Attributes plans constructed in terms of unity value

In the ISO 2859-1 standard, the plans are constructed in such a manner as to have constant acceptance number values across the diagonals of the sampling plan tables. This section provides a short rationale for this approach.

ISO 2859-1, the AQL (PRQ) values and the sample size values are "approximate" geometric series. The following table shows a selection of sample size values along with the ratio between consecutive values.

Table 1: Sample size values from ISO 2859-1 as a geometric series

Sample size	Ratio between consecutive sample sizes
5	-
8	1.60
13	1.63
20	1.54
32	1.60
50	1.56

As can be seen, the ratio between two consecutive sample size values is always close to 1.6. The ratio between consecutive AQL (PRQ) values is also approximately 1.6, as shown in the following table.

Table 2: AQL (PRQ) values from ISO 2859-1 as a geometric series

AQL	Ratio between consecutive AQL values
0.010	-
0.015	1.50
0.025	1.67
0.040	1.60
0.065	1.63
0.100	1.54

As a result, the product AQL (PRQ) × sample size remains "near constant" across the diagonals of the sampling plan tables. This is illustrated in the following table, for a selection of AQL values.

Table 3: The product PRQ × sample size remains "near constant" across the diagonals of the sampling plan tables

Sample size	AQL (PRQ)										
	0.001	**0.0015**	**0.0025**	**0.004**	**0.0065**	**0.01**	**0.015**	**0.025**	**0.04**	**0.065**	**0.1**
2	0.002	0.003	0.005	0.008	0.013	0.02	0.03	0.05	0.08	0.13	0.2
3	0.003	0.005	0.008	0.012	0.020	0.03	0.05	0.08	0.12	0.20	0.3
5	0.005	0.008	0.013	0.020	0.033	0.05	0.08	0.13	0.20	0.33	0.5
8	0.008	0.012	0.020	0.032	0.052	0.08	0.12	0.20	0.32	0.52	0.8
13	0.013	0.020	0.033	0.052	0.085	0.13	0.20	0.33	0.52	0.85	1.3
20	0.020	0.030	0.050	0.080	0.130	0.20	0.30	0.50	0.80	1.30	2.0
32	0.032	0.048	0.080	0.128	0.208	0.32	0.48	0.80	1.28	2.08	3.2
50	0.050	0.075	0.125	0.200	0.325	0.50	0.75	1.25	2.00	3.25	5.0
80	0.080	0.120	0.200	0.320	0.520	0.80	1.20	2.00	3.20	5.20	8.0
125	0.125	0.188	0.313	0.500	0.813	1.25	1.88	3.13	5.00	8.13	12.5
200	0.200	0.300	0.500	0.800	1.300	2.00	3.00	5.00	8.00	13.00	20.0
315	0.315	0.473	0.788	1.260	2.048	3.15	4.73	7.88	12.60	20.48	31.5
500	0.500	0.750	1.250	2.000	3.250	5.00	7.50	12.50	20.00	32.50	50.0
800	0.800	1.200	2.000	3.200	5.200	8.00	12.00	20.00	32.00	52.00	80.0
1250	1.250	1.875	3.125	5.000	8.125	12.50	18.75	31.25	50.00	81.25	125.0
2000	2.000	3.000	5.000	8.000	13.000	20.00	30.00	50.00	80.00	130.00	200.0

The product AQL (PRQ) × sample size is called the *unity value* and can be understood as the number of expected nonconforming items in the sample for lot quality AQL. For example, for lot quality 1% percentage nonconforming and a sample size of 20 items, we can expect 0.2 nonconforming items. This is the rationale for having constant acceptance number values across diagonals in ISO 2859-1.

5.2.2 Variables plans constructed in terms of the producer's risk

The "philosophy" of ISO acceptance sampling plans for inspection by variables is as follows.

First, the ISO plans are designed in such a manner as to ensure either a high probability of acceptance at the acceptance quality limit (AQL) i.e. at the producer's risk quality level (PRQ) or a low probability of acceptance at the limiting quality (LQ) i.e. at the consumer's risk quality level (CRQ).

Secondly, the ISO plans indexed by AQL are constructed in such a way that the producer's risk decreases as the lot size increases. The following table, taken from the Mathematical and Statistical Principles underlying Military Standard 414 [30], the forerunner of the ISO 3951 standard, shows the producer's risk in terms of the sample size code letter (reflecting lot size):

Table 4: Lot size in the ISO standards

Sample size code letter	Producer's risk
B	0.11
C	0.10
D	0.10
E	0.10
F	0.10
G	0.09
H	0.08
I	0.07
J	0.06
K	0.06
L	0.05
M	0.05
N	0.04
O	0.03
P	0.02
Q	0.01

As can be seen the "target" PR of 5 % is only achieved from code letter L onwards. Indeed, the PR is better than 5 % from code letter N onwards, achieving 1 % for code letter Q.

In the plans in ISO 3951-2, the producer's risk remains "near constant" along the diagonals (from bottom left to top right).

The principle behind the ISO 3951-6 plans (which are indexed by LQ) is different: here the aim is to design plans whose OC curves correspond to the OC curves in ISO 2859-2.

5.3 Acceptance sampling versus conformity assessment

There is an extensive normative body of work on conformity assessment: the ISO 17000 series, JCGM 106, etc. The question thus arises to what extent this normative literature is relevant for acceptance sampling. In particular, the question arises whether conformity assessment procedures can be used in acceptance sampling.

In this section, the following abbreviations will be used:

- AS = acceptance sampling
- CA = conformity assessment

It is important to note that the question addressed here cannot currently be answered definitively one way or another. This section can thus be considered to provide basic orientation and considerations which may prove useful in untangling these various concepts in a given context.

5.3.1 Definitions

JGCM 106

In JCGM 106, conformity assessment is defined (definition 3.3.1) as

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

Note that this definition is so general as to allow lot inspection to fall under its scope. Indeed, in many cases a lot can be considered the product output of a process.

ISO 3534

In ISO 3534-2, in section 4 Inspection and general acceptance sampling, we find definition 4.1.1

> *Conformity evaluation*
>
> *Systematic examination of the extent to which an item/entity fulfills specific requirements.*

If conformity evaluation is taken as synonymous with conformity assessment, then the fact this definition is found in the section on acceptance sampling is an indication that the CA normative literature is indeed relevant for AS.

ISO 17025

It seems useful to recall the definition of a “decision rule” (for use in conformity assessment) in ISO 17025. (This definition highlights the central role which measurement uncertainty plays in conformity assessment.)

> Rule that describes how measurement uncertainty is accounted for when stating conformity with a specified requirement.

5.3.2 Positions in ISO standards

ISO 10576 (Guidelines for the evaluation of conformity with specified requirements)

On the other hand, the following paragraph from ISO 10576 would seem to indicate that a resounding no is the correct answer:

> *Because of the apparent similarity to acceptance sampling procedures, it is sometimes seen that acceptance sampling plans are used in conformity testing activities. Acceptance sampling and conformity testing activities both utilize elements of hypothesis testing (see e.g. ISO 2854). It is, however, important to realize that the objectives of the two activities are fundamentally different and in particular the two activities imply different approaches to the risk involved (see ISO 2854 and Holst).*

ISO 2859 and ISO 3951

In the standards from both series, the following phrase is found in the forward:

> *For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO’s adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: http://www.iso.org/iso/home/standards_development/resources-for-technical-work/foreword.htm*

This phrase seems to imply that there is a connection between acceptance sampling and conformity assessment.

5.3.3 Positions in the literature

Paper by Holst, Thyregod and Wilrich (On conformity testing and the use of two stage procedures)

This paper draws the following distinction:

- Acceptance sampling plans are used in the context of transactions between two parties and should provide unambiguous rules for accepting or rejecting the lot. Both parties are aware of the risks involved.
- On the other hand, in conformity testing, “it is crucial that the user can have confidence in a declaration of conformity”. Thus, when an item meets the conformity criterion, this means that “the test has demonstrated beyond any reasonable doubt that the entity conforms to the requirements.”

5.3.4 Discussion

The following distinctions between CA and AS seem to be clear:

- in CA, testing is performed on the basis of one single item, and measurement uncertainty is taken into account
- in AS, there are many cases where testing is performed on the basis of several items (sampled from the lot). In inspection by variables, it is not the conformity of each item which is determined, but rather, one test result is obtained per item and the decision to accept or reject the lot is made on the basis of calculations performed on these test results.
- The outcome of CA may be inconclusive whereas in AS there is always an acceptance or rejection outcome.

A related difference between the two is as follows:

- In CA, measurement uncertainty is taken into account in the decision rule. Thus, in CA, the focus is on the measurand (in the strict metrological sense).
- In AS, the rule for lot acceptance or rejection takes into account the lot standard deviation, which describes how the property of interest varies in the lot, rather than variation between *test results*, which may reflect other effects such as analytical uncertainty, effects due to the sampling procedure, etc. Thus, in AS, the acceptance rule is expressed in terms of the statistical properties of the lot.

The following points highlight conceptual similarities between conformity assessment and acceptance sampling:

- Acceptance sampling can be “re-interpreted” in such a way that the entire framework is formulated in terms of a “measurand” – thus achieving a common conceptual framework with conformity assessment. In this re-interpretation it is the statistical parameters of the lot (e.g. lot mean and lot standard deviation) which constitute the measurand. See Uhlig et al. (2022) [28].
- In the classical CA framework, conformity often requires the measurement uncertainty to be sufficiently low, e.g. in the case of a decision rule such as $y_m + U < USL$. Similarly, in AS, one could formulate requirements regarding sufficiently low specific producer or consumer risks.
- for both CA and AS, one can define both “parametric” and “specific” risks (see section 6.5.1).

5.4 The role of measurement uncertainty in acceptance sampling

The criterion for lot acceptance or rejection is often expressed in terms of statistical parameters such as lot mean and lot standard deviation. If the measurement uncertainty is non-negligible, then the estimates of these statistical parameters may be affected. Accordingly, in some cases, it may be appropriate to apply a correction for measurement uncertainty. Naturally, such a correction presupposes that a reliable estimate of measurement uncertainty is available.

The reader is referred to the *Guidelines on measurement uncertainty* (CXG 54-2004) and the information document for information about the estimation of measurement uncertainty; another key reference is ISO 5725 Parts 1 & 2.

The Eurachem Guide on *Measurement Uncertainty arising from sampling* provides guidance regarding the estimation of the sampling component of uncertainty. The duplicate method can also be applied to estimate the lot standard deviation. Such a method would be appropriate if within-item variation is expected. If an estimate of repeatability precision is available, then Hahn’s method (see Section 4.2.1 and Section 4.2.2) can be used to adjust the estimate of the lot standard deviation, avoiding the need to test items in duplicate. The Eurachem Guide also provides information regarding the use of control charts for the monitoring of consistency.

The following questions are discussed in this section:

- How is the measurand specified?
- Terminological clarification: sampling uncertainty versus acceptance sampling
- What effect does analytical uncertainty have on the producer’s risk?

5.4.1 Specifying the measurand

In determining measurement uncertainty, the first question is: *what is the measurand?*

The term *measurand* has a very specific definition in metrology. The full definition ("*Quantity intended to be measured*") can be found in VIM (JCGM 200:2012). The definition of *quantity* is rather technical. For our purposes here, it is sufficient to highlight 2 aspects of the definition of *measurand*.

In order to specify a measurand, it is necessary to define both

- the property of interest (e.g. analyte concentration)
- in what material/sampling target[6] this property of interest is being measured

For instance: measuring a given analyte concentration *in an individual item* and measuring the mean analyte concentration *in the lot* correspond to two different measurands.

It should also be noted that a measurand is by definition a property whose characterization is quantitative rather than qualitative.

The question which sources of uncertainty are relevant is answered by considering the definition of the measurand. For instance, if the measurand is defined in terms of the laboratory sample, then only analytical sources are relevant. However, if the measurand is defined in terms of a population/lot/container ("sampling target") from which the laboratory sample was obtained, then both sampling and analytical sources are relevant.

In connection with acceptance sampling, the concept of measurand can be understood in two different ways.

5.4.1.1 Classical definition of measurand

Insofar as test results are obtained (whether on the basis of discrete items, or on the basis of a composite sample), these test results involve the specification of a measurand. The question arises whether the measurand is specified in relation to the laboratory sample, or in relation to the lot. Two separate cases must be considered: lots consisting of discrete items and lots consisting of bulk material.

Lots consisting of discrete items

In the case of lots consisting of discrete items, acceptance is often based on a characterization of percentage nonconforming. The acceptance criterion is expressed in terms of the lot standard deviation (estimated from the item-specific test results) and the mean value across items. For a given item, the aim is to characterize the item-specific mean value—not the lot mean. Accordingly, the measurand is defined in relation to the laboratory sample[7], and only analytical sources of measurement uncertainty need be considered. *In particular, there is no sampling component of measurement uncertainty.*

Note: the mean value across the item-specific test results may be considered an estimate of the lot mean. Nonetheless, for lots consisting of discrete items, the measurand is the item-specific mean—not the lot mean.

Lots consisting of bulk material

By contrast, in the case of bulk materials, the aim is to obtain an estimate of the mean concentration in the lot. Accordingly, the measurand is specified in terms of the lot, *and both analytical and sampling sources of uncertainty apply.*

5.4.1.2 Reinterpretation of the concept of measurand for acceptance sampling

Insofar as acceptance is based on a criterion expressed in terms of the statistical parameters of the lot under inspection, it is useful to take a step back and to generalize the concept of measurand as follows:

- In acceptance sampling, the measurand can be defined in terms of the statistical parameters of the lot (e.g. for a lot consisting of discrete items, the lot mean and the lot standard deviation).
- The measurement uncertainty can then be considered to reflect the statistical uncertainty of the estimates of these parameters.
- This reinterpretation is particularly relevant in connection with Bayesian approaches to acceptance sampling. See Uhlig et al. (2022) [28].

5.4.2 Sampling uncertainty versus acceptance sampling (terminological clarification)

Sampling uncertainty (Uncertainty from Sampling, UfS)

[6] A sampling target is defined in Eurachem UfS Guide (2019) as *the portion of material, at a particular time, that the sample is intended to represent.* The reader is also referred to AMC(2005), Technical Brief 19.

[7] A given laboratory sample may correspond to an entire item or may correspond to a subsample from the item. In the latter case, if the measurand is defined in relation to the item, there may be a within-item sampling component of measurement uncertainty.

Sampling uncertainty is a component of measurement uncertainty.

If the measurand is specified in terms of a larger population such as a lot/container/area, then the laboratory sample must be considered the result of a sampling procedure which may contribute to the uncertainty of the test result. The larger population from which the laboratory sample was obtained is often referred to as the sampling target.

If the total measurement uncertainty is too large, it may be necessary to improve the sampling procedure.

If the measurand is specified in terms of the laboratory sample, there is no contribution to measurement uncertainty due to sampling.

Acceptance sampling

In acceptance sampling, the aim is not to obtain an estimate of measurement uncertainty. The only connection between acceptance sampling and measurement uncertainty is the possible effect of the latter on the calculation of statistical parameters such as lot mean value and lot standard deviation in terms of which the acceptance criterion is expressed.

Lot standard deviation versus sampling component of measurement uncertainty

The concept of the lot standard deviation may seem to be closely related to the concept of the sampling component of measurement uncertainty, namely as a measure of the variation of the property of interest within the lot (where the lot is interpreted as a sampling target). However, this similarity is merely superficial; at a more fundamental level, the two concepts must be carefully distinguished.

In order to clarify the distinction between the lot SD and the sampling component of measurement uncertainty, consider the following hypothetical scenario: if all items in the lot were tested then there would be no sampling component of the uncertainty of the lot mean (assuming homogeneity within the items). Nonetheless, variation between the items could be small or large.

Note regarding lots consisting of bulk material

For lots consisting of bulk material, the acceptance criterion is often expressed in terms of the lot mean (rather than in terms of the proportion non-conforming). The estimate of the lot mean can be obtained from a composite sample. If the acceptance criterion also involves the uncertainty of the lot mean, and the calculation of this uncertainty includes contributions reflecting the sampling procedure, then this procedure may be indistinguishable from the procedures typically applied in connection with conformity assessment. In such cases, there may thus be no useful distinction between acceptance sampling and conformity assessment. In particular, in such cases, measurement uncertainty takes on a completely different role: rather than a “nuisance parameter” which must be corrected for (if non-negligible), it now plays a central role.

5.4.3 Effects of analytical and sampling uncertainty in acceptance sampling

In this section, we consider the case that the lot consists of discrete items. The criterion for lot acceptance is thus expressed in terms of the percentage nonconforming.

Notation: let n denote the sample size (i.e. the sample consists of n items). For item i, the corresponding test result is denoted x_i. Proposed model for test result x_i:

$$x_i = \mu_i + B + e_i$$

where

μ_i is the “true” mean value for item i. The “true” lot standard deviation σ characterizes the variation of the μ_i in the lot. If sigma is unknown, it is estimated on the basis of the x_i values obtained from the items in the sample.

B is the bias (systematic effect). First and foremost, this term reflects laboratory bias or analytical method bias, but there may be other contributions to bias, e.g. from the sampling procedure.

e_i is the random effect for item i. First and foremost, this term reflects analytical measurement uncertainty (repeatability effects), but there may be contributions from sampling uncertainty.

For lots consisting of discrete items, the acceptance criterion typically has the following form:

$$\bar{x} + ks \leq U \text{ (or } \bar{x} \leq A \coloneqq U - ks)$$

where

$\bar{x}$ is the mean value across the item-specific test results $\bar{x} = \frac{1}{n}\sum_{i=1}^{n} x_i$

s is the standard deviation across the item-specific test results $s^2 = \frac{1}{n-1}\sum_{i=1}^{n}(x_i - \bar{x})^2$

Analytical uncertainty will manifest itself in the term $e_i \sim \mathcal{N}(0, u_{analytical})$ and will always **inflate** the estimate of the lot standard deviation s:

$$s^2 = \sigma^2 + u_{analytical}^2$$

where

σ is the "true" lot standard deviation

$u_{analytical}$ is the analytical measurement uncertainty

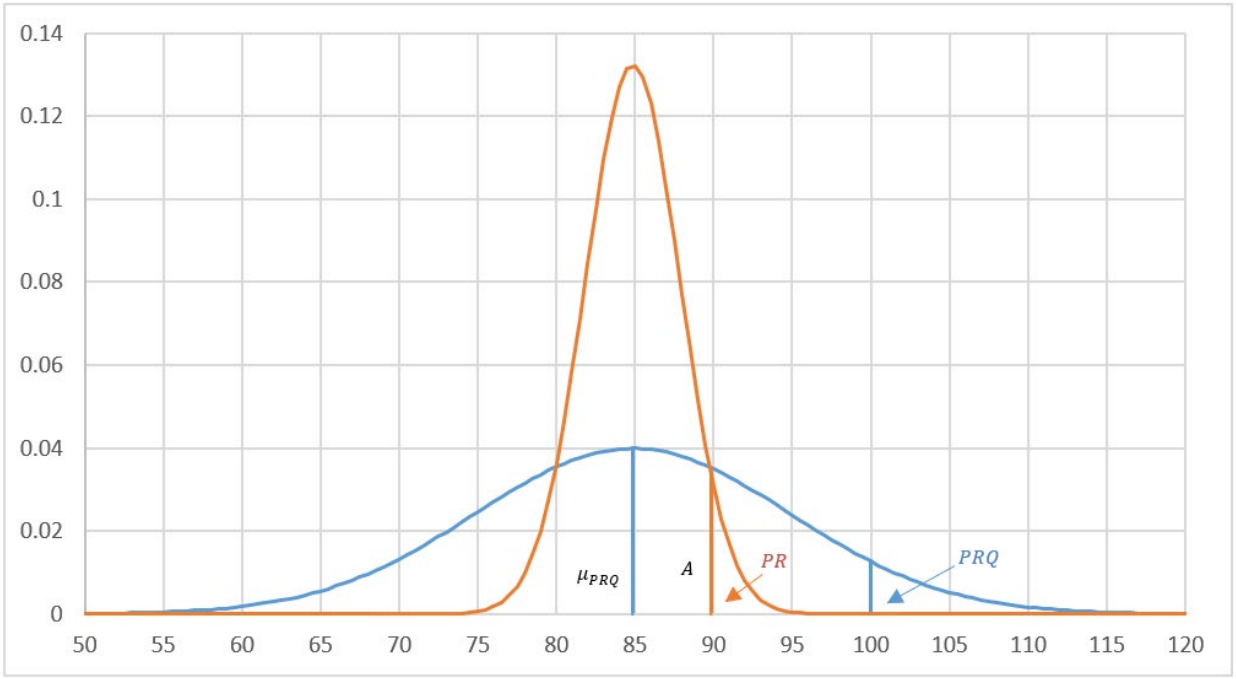
If left uncorrected, the presence of analytical uncertainty will increase producer and consumer risks.

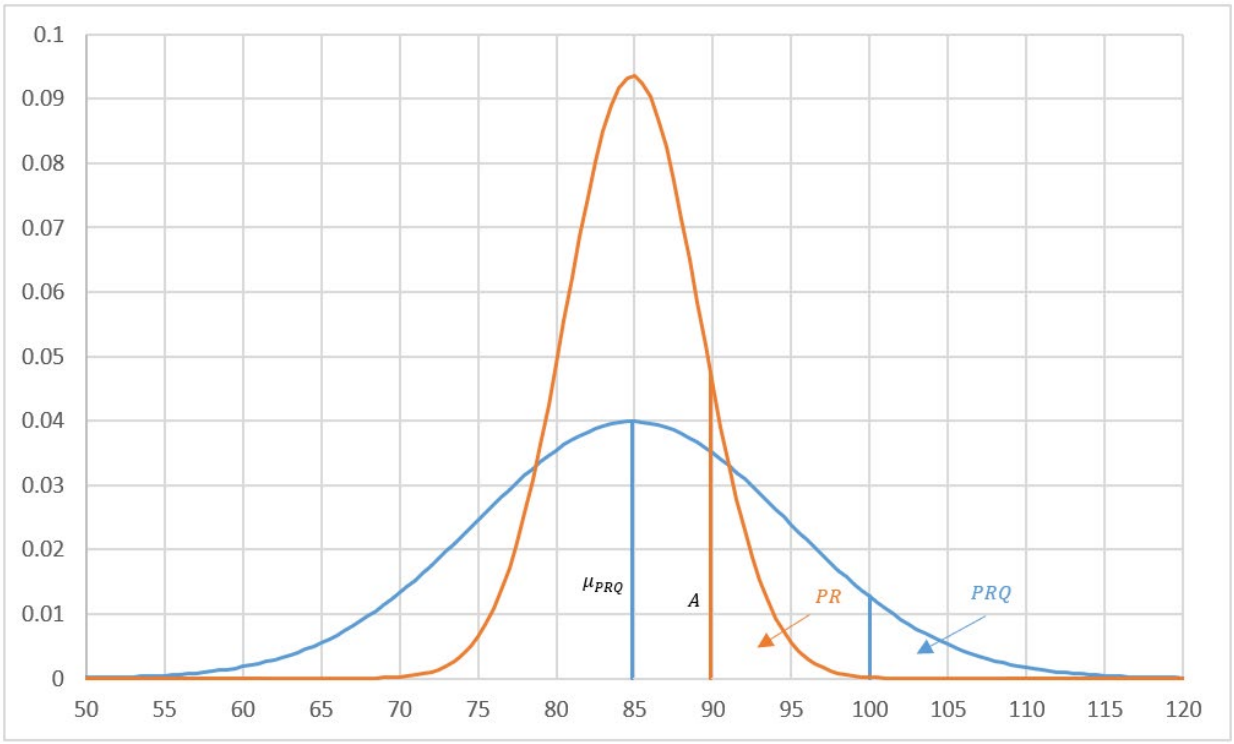
This is illustrated on the basis of two scenarios.

Scenario 1: known σ, increase in producer risk

In this scenario, the upper specification limit is $U = 100$ and the lot standard deviation $\sigma = 10$ is known. The following acceptance sampling plan is applied:$n = 11$, $k = 1.025$ ($A = U - k \cdot \sigma = 90$). However, the analytical uncertainty is nonnegligible, with $u_{analytical} = 10$ (the analytical uncertainty is the equal to the lot SD, and hence can be considered considerable). As a result of the analytical uncertainty, for a lot with quality $PRQ = 6.5\%$, the PR is over 11% (instead of 5%) due to the inflated variation of the x_i.

Figure 36: The blue curve represents the distribution of the property of interest in the lot. The area of the blue curve above $U = 100$ is $PRQ = 6.5\%$. The red curve represents the statistical distribution of the arithmetic mean. In the top diagram, there is no analytical uncertainty, so the PR is 5%. In the bottom diagram, the analytical uncertainty is non-negligible, resulting in an increase in PR to over 11%.

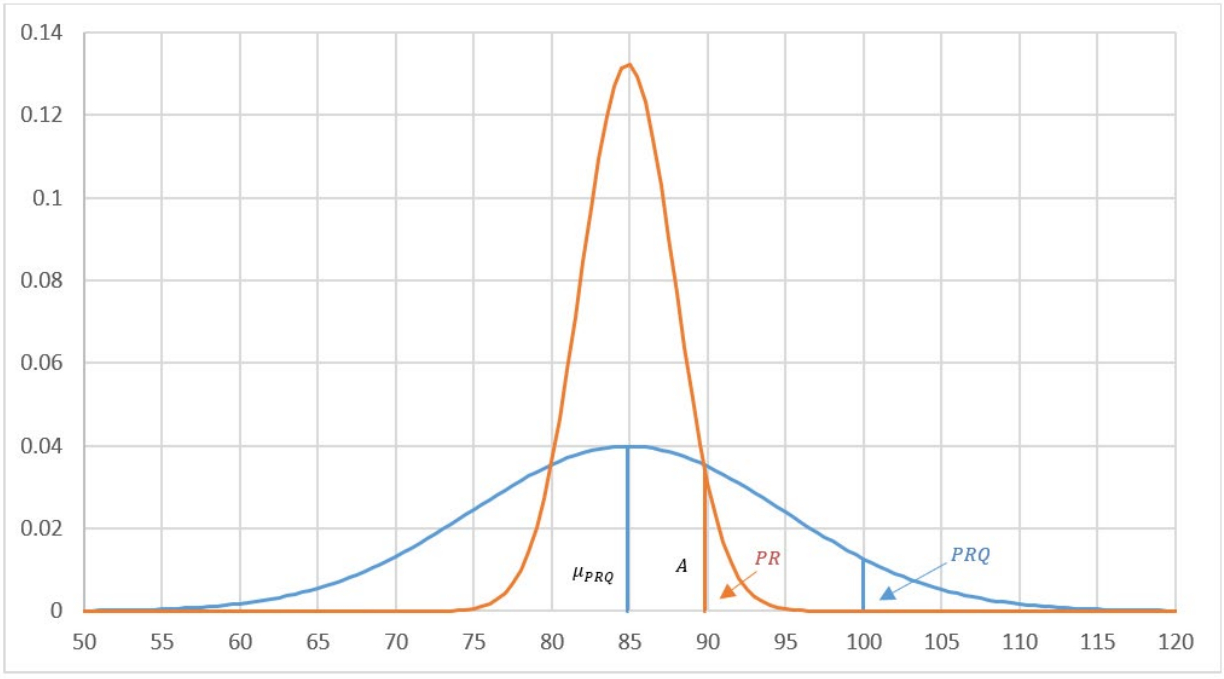


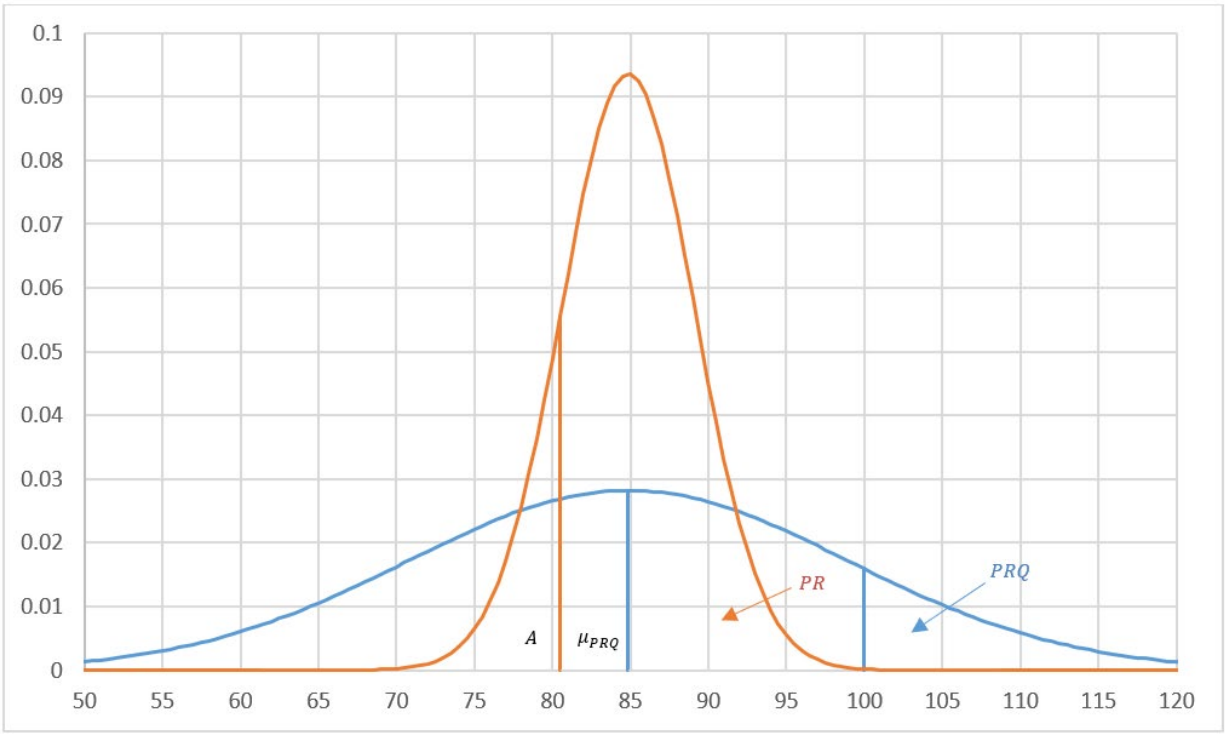


Scenario 2

In this scenario, the upper specification limit is $U = 100$ and the lot standard deviation is unknown and estimated from the x_i. At this point, various things can happen. For instance, the producer could notice that the lot quality is now 8.5 % percentage nonconforming instead of 6.5 %. If this discrepancy is ignored, and the same plan as originally contemplated is applied (in particular: $k = 1.025$), the acceptance limit is now 80.5 (instead of 90) due to the inflated estimate s, and the PR is now over 85 %.

Figure 37: The blue curve represents the distribution of the property of interest in the lot. The area of the blue curve above $U = 100$ is $PRQ = 6.5\%$. The red curve represents the statistical distribution of the arithmetic mean. In the top diagram, there is no analytical uncertainty, so the PR is 5%. In the bottom diagram, the analytical uncertainty is non-negligible, resulting in an increase in a much wider blue curve and a distorted value for A, resulting in a PR of nearly 85 %.

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PART THREE

Bayesian Sampling Plans

6 Bayesian plans

This section presents an overview on Bayesian plans that are the subject of new and on-going work in ISO and other forums.

It is often the case that prior information regarding lot quality is available. For instance, the consumer may have previously purchased lots from the producer of the lot currently under inspection. It is thus sensible to ask the following question: is it possible to propose a Bayesian framework for the design of acceptance sampling plans which mobilizes prior information in order to achieve a reduction in sample size? In the following, this question will be discussed in relation to *inspection by attributes*.

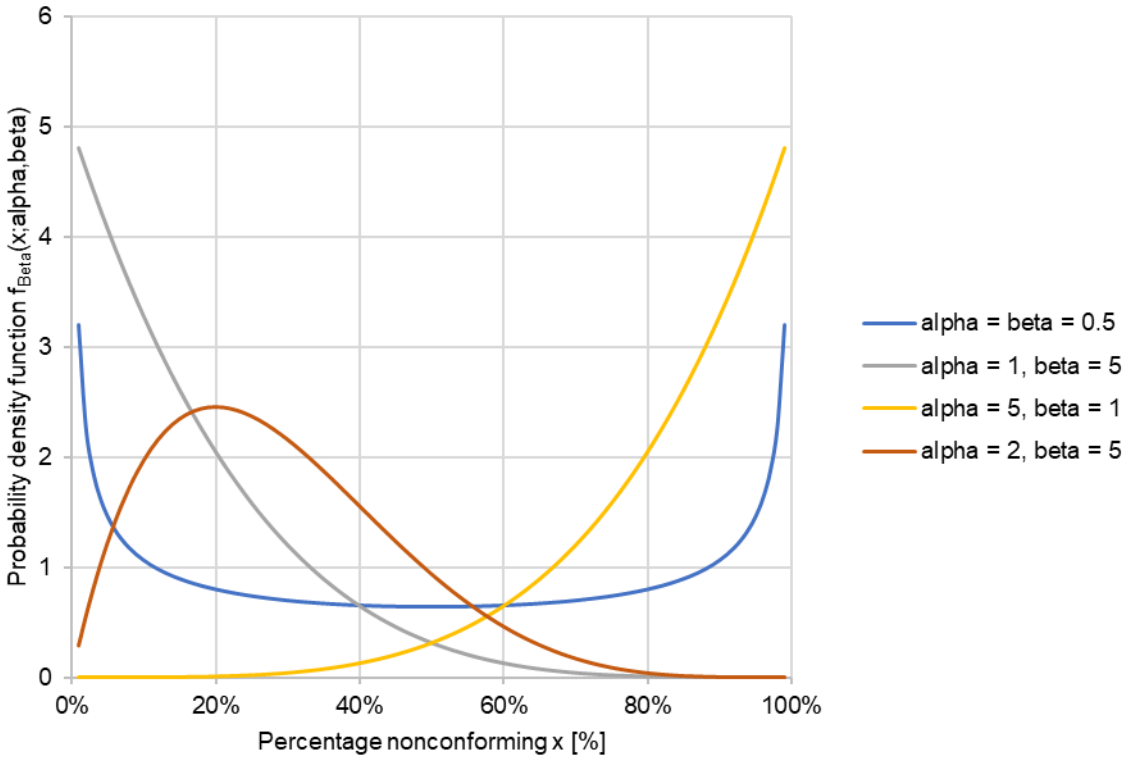
6.1 Prior distributions

The consumer's or producer's prior information regarding the percentage nonconforming x is encapsulated in the prior distribution. In the case of inspection by attributes, the simplest assumption regarding the prior for x is that it follows a beta distribution.

The beta family of distributions is generated via two hyperparameters α and β. For a given choice of α and β, the corresponding beta distribution is denoted Beta(α, β) and the probability density function is denoted $f_{Beta}(x; \alpha, \beta)$.

The following diagram shows different beta distributions. As can be seen, this family of distributions is quite versatile, allowing very different curves to be mapped via the choice of α and β.

Figure 38 Different beta distributions



Note: The case that no prior information is available can be represented by the choice $\alpha = \beta = 0.5$.

Once items from a lot have been tested, the prior distribution can be updated to obtain a posterior distribution.

If the prior is a beta distribution, the posterior is also a beta distribution and the posterior hyperparameters α_1 and β_1 are obtained from the prior hyperparameters α_0 and β_0 and from the number of nonconforming items y as follows:

$$\alpha_1 = \alpha_0 + y$$

$$\beta_1 = \beta_0 + n - y$$

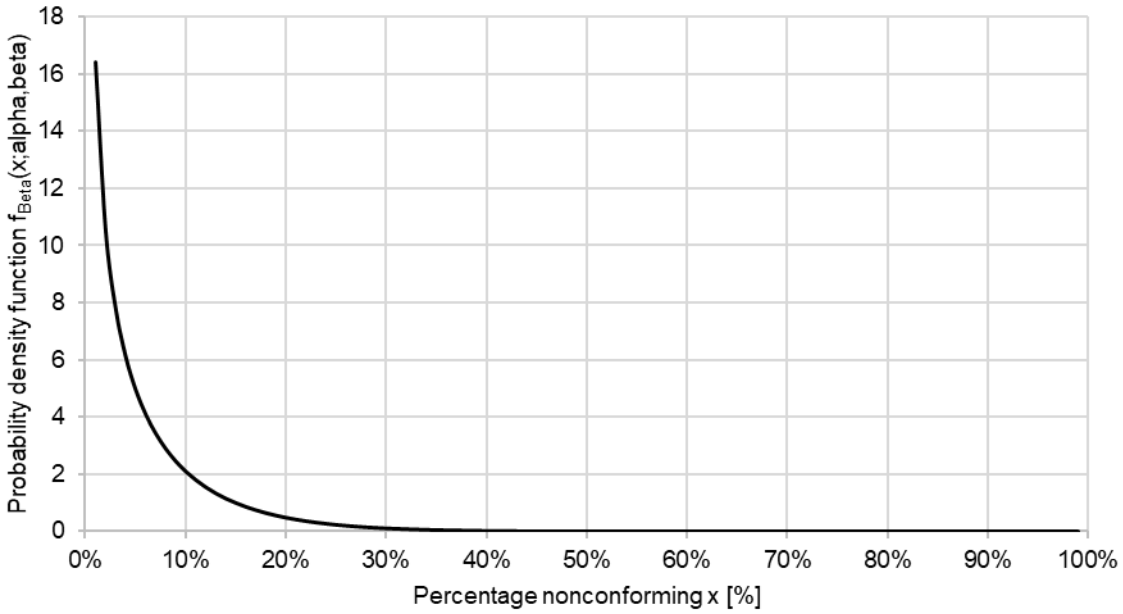
For example, if 10 items from a given lot have been inspected and all 10 have been found to be conforming (i.e. $y = 0$), the $\alpha_0 = \beta_0 = 0.5$ prior is updated as follows:

$$\alpha_1 = 0.5 + 0 = 0.5$$

$$\beta_1 = 0.5 + 10 - 0 = 10.5$$

The following diagrams shows the beta distribution corresponding to $\alpha = 0.5$ and $\beta = 10.5$.

Figure 39 Probability density for Beta distribution α=0.5, ß= 10.5



As can be seen, the Beta(0.5,10.5) density function falls steeply in the range $x = 0\%$ to $x = 30\%$ and is near 0 for all percentage nonconforming values above $x = 30\%$. This means that—on the basis of the prior Beta(0.5,0.5) and the test outcome $y = 0$ (out of 10 tests)—it is now expected that the percentage nonconforming will be no greater than 30%.

6.2 Conformance probability approach

In this section, an approach is presented for specifying acceptance sampling plans on the basis of the concept of conformance probability from JCGM 106. The approach described in the ISO 2859 and ISO 3951 standards asks the following question: given a certain quality level (expressed e.g. as percentage nonconforming) what is the probability that the lot is accepted? By contrast, the conformance probability approach asks the following question: given that a lot is accepted, what is the probability that it is actually conforming? Insofar as the conformance probability approach starts from lot acceptance or rejection (i.e. information which is known), this approach can be considered more pragmatic. In the conformance probability approach, probabilities and risks are calculated via the Bayesian approach. The starting point is a prior distribution, which encapsulates all available knowledge regarding the property of interest prior to lot inspection. Once tests have been performed, the prior distribution is updated on the basis of the test results. The updated distribution is called the posterior distribution.

Definition of conformance probability

The definition of conformance probability found in JCGM 106 can be adapted to lot inspection and acceptance sampling as follows.

Conformance probability is the probability that the lot quality actually lies in the conformance region $\mathcal{C}$. This probability is calculated on the basis of the *posterior* distribution.

As can be seen from the definition, a conformance region for lot quality must be specified. This is a clear departure from the approach described in the ISO 2859 and ISO 3951 standards in which, even though the plans are indexed in terms of certain quality levels considered “good” (PRQ, AQL) or “poor” (CRQ, LQ), conformance regions for lot quality are not specified.

The conformance region $\mathcal{C}$ can be specified via an upper limit for the percentage nonconforming. This upper limit is denoted $x_{\mathcal{C}}$.

Definition of parametric and specific consumer’s and producer’s risks

In the ISO 2859 and ISO 3951 standards., the consumer and producer risks are defined as follows:

- Producer risk = probability that a lot of good quality (e.g. PRQ or AQL) will not be accepted
- Consumer risk = probability that a lot of poor quality (e.g. CRQ or LQ) will be accepted

In the calculation of the ISO risks, lot quality is treated as the parameter of a statistical distribution. For this reason, the ISO risks are also called “parametric” risks.

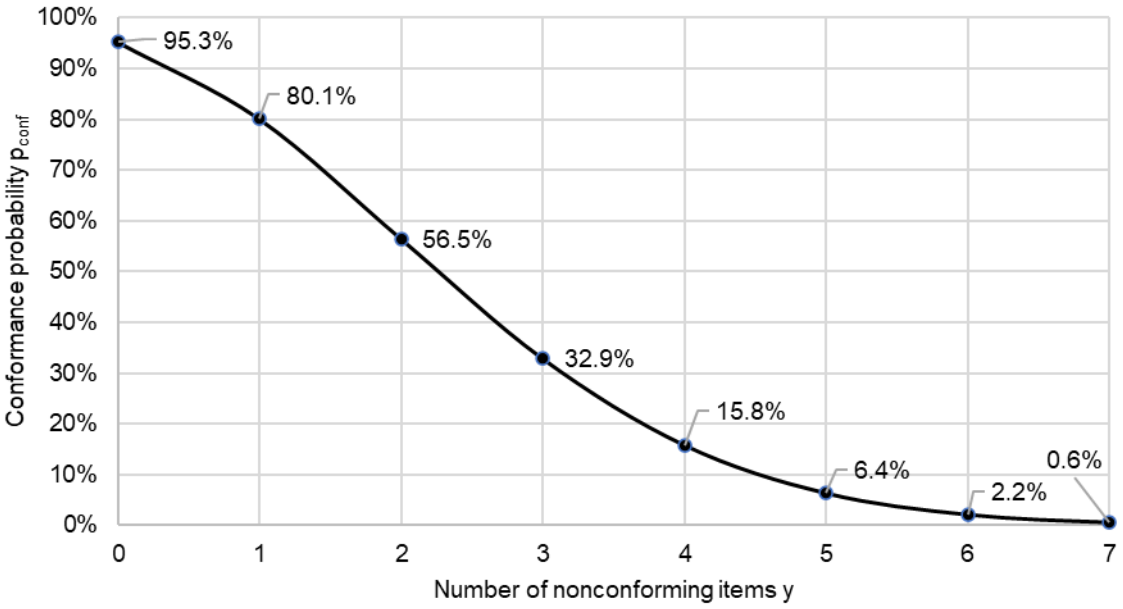
In JCGM 106, specific consumer and producer risks are defined by “going in the opposite direction” as compared to the ISO definitions:

- Specific producer risk = probability that a lot *which has not been accepted* actually has conforming quality
- Specific consumer risk = probability that a lot *which has been accepted* actually has nonconforming quality

Conformance probability curves

The concept of conformance probability can be used to derive acceptance sampling plans. For a given sample size n, the conformance probability is calculated for each possible testing outcome y (e.g. number of nonconforming items in the sample). An acceptance sampling plan can then be determined by requiring that the specific consumer risk (the complement of the conformance probability) should be less than a certain threshold (e.g. 5 %). This procedure is illustrated in the following diagram which shows the conformance probability curve for a lot conformity region specified via the upper limit $x_c = 10\%$ for the percentage nonconforming, the prior Beta(1,9) and the sample size $n = 20$.

Figure 40: Conformance probability curve for upper limit of the lot conformity region $x_c = 10\%$, prior Beta(1,9) and sample size $n = 20$



As can be seen, the conformance probability is greater than 95 % at $y = 0$. Accordingly, the plan $n = 20$, $c = 0$ is a plan which allows a specific consumer risk less than 5 %.

Consumer and producer risks

The Bayesian framework from JCGM 106 allows the definition of various risks. These risks address different questions, as summarized in the following tables.

Table 5: Bayesian producer’s risks – notation and interpretation

Risk	Notation	Definition
Specific PR (evaluated for a specific test outcome y resulting in rejection)	SPR(y)	How likely is it that a lot is conforming, given that it is rejected?
Conditional PR (conditioned on the lot quality x)	CPR_x	How likely is it that a lot is rejected, given that it is conforming?

Risk	Notation	Definition
Conditional PR (conditioned on all test outcomes y resulting in rejection)	CPR_y	How likely is it that a lot is conforming, given that it is rejected?
Global PR	GPR	How likely is it that a lot is both conforming and rejected?
Global probability of rejection	GP_{rej}	How likely is it that a lot is rejected – no matter whether it is conforming or not?

Table 6: Bayesian consumer's risks – notation and interpretation

Risk	Notation	Interpretation
Specific CR (evaluated for a specific test outcome y resulting in acceptance)	$SCR(y)$	How likely is it that a lot is nonconforming, given that it is accepted?
Conditional CR (conditioned on the lot quality x)	CCR_x	How likely is it that a lot is accepted, given that it is nonconforming?
Conditional CR (conditioned on all test outcomes y resulting in acceptance)	CCR_y	How likely is it that a lot is nonconforming, given that it is accepted?
Global CR	GCR	How likely is it that a lot is both nonconforming and accepted?
Global probability of acceptance	GP_{acc}	How likely is it that a lot is accepted – no matter whether it is conforming or not?

More information regarding the conformance probability approach can be found in Uhlig et al. (2024) [21].

6.3 Utility approach

6.3.1 Definition

The definition of utility is as follows:

Utility for a lot which has been **accepted**	=	benefit associated with an accepted lot (under the assumption that all items are conforming) i.e. returns minus expenditures
		minus damages associated with nonconforming items in an accepted lot
		minus testing and sampling costs
Utility for a lot which has been **rejected**	=	**minus** testing and sampling costs

Depending on the context and the consumer, benefits may reflect profits from commercial sales, tax income, long-term environmental considerations, laying the foundations of new business opportunities, etc.; while damages may reflect commercial losses, negative health impacts, costs associated with recalling a lot and a tarnished public image.

6.3.2 The consumer

In acceptance sampling, the consumer is defined as the party which accepts or rejects the producer's lot. The consumer may be a retailer purchasing commodities at a wholesale market, a manufacturer acquiring parts or a customs officer or food safety agent checking that legal limits for contaminants are not exceeded before admitting a lot at the border, etc... The following table provides different interpretations of benefit and damages for three different types of consumers.

Table 7: The meaning of benefit and damages for different types of consumers

	Retailer	**Manufacturer**	**State**
Benefit associated with conforming items	Profit = *returns* minus *expenditures* of conforming items *Returns* = income from selling the items in the lot *Expenditures* = the lot's purchase price, transport costs, staff remuneration, retail outlet overhead, taxes	Profit from conforming items depends on aspects like cost efficiency, innovation, secure supply, flexibility, and sustainability	Benefit from conforming items could include: Positive impact on trade balance Benefits for the end-user Employment impact Tax revenue Innovation Positive environmental impact Positive health impact
Damages associated with nonconforming items	Income loss associated with nonconforming items, e.g. with items which were not sold, contractual penalties or legal fines, costs associated with containing and recalling items	Income loss from nonconforming items, associated with disruptions in the manufacturing process, quality issues and supply chain challenges	Damages from nonconforming items could include Negative health impact Loss in GDP Negative employment impact Financial losses (e.g. tax, extra expenditures) Negative environmental impact

6.3.3 Mathematical expression

The definition of utility makes it possible to apply a simple criterion in the design of acceptance sampling plans: select the plan which maximizes utility.

In order to express the utility function in mathematical terms, the following notation will be used:

B = benefit associated with one conforming item in an accepted lot

D = damages associated with one nonconforming item in an accepted lot (damages is understood here in a very general sense, see below. This parameter could also be called "losses".)

T = the testing and sampling costs per item

All costs are expressed in terms B (the benefit associated with a conforming item in an accepted lot). In other words, the common unit in which all the terms in the utility function are expressed is B. For example, the damages associated with a nonconforming item D could be 10 B; and the testing and sampling costs per item T could be 5 B. The values for D and T expressed in terms of B will be referred to the *cost structure*.

The quantity B thus has a dual role: on the one hand, it denotes the item-specific benefit and on the other hand, it functions as a unit in which to express D (damages) and T (testing costs). The values for D and T are thus *relative* to B. The advantages of expressing D and T in relation to B are twofold:

- on the one hand, it may be easier to provide values for D and T in relation to B rather than monetary values

- on the other hand, the fact that these values are relative highlights that it is the relation between the costs and benefits rather than the absolute or monetary values themselves which play a central role in the utility approach

Specifying the parameter B point of view of a state (the consumer is e.g. a food safety agency or a customs officer) is not always straightforward. Indeed, from the point of view of the state, some of the positive aspects of a successful commercial transaction may be intangible or difficult to assign a precise monetary value to. In such cases, as a rule of thumb, it is suggested to use the lot's purchase price.

The damages (or losses) parameter D can have different meanings. If there are no other costs and if there are no re-purposing options for items which are not sold, the value $D = B$ corresponds to the case that the only cost associated with a nonconforming item is the *loss* in income caused its not being sold. A value such as $D = 1.5\,B$ corresponds to the scenario that, in addition to losses, there are additional costs associated with nonconforming items, such as costs associated with waste management. A value such as $D = 10\,B$ could reflect additional costs associated with environmental pollution, damage to the reputation of the retailer (dissatisfied customers taking their business elsewhere, tarnished public image) etc. Higher values such as $D = 25\,B$ or $D = 100\,B$ reflect substantial additional costs such as those associated with recalling a lot or healthcare costs. It may be difficult to quantify healthcare costs associated with nonconforming items (e.g. hospitalization costs due to the ingestion of contaminated meat). One approach could be to introduce an auxiliary unit such as W = wages corresponding to a day's work for an "average worker." For example, if a brief stay in a hospital is quantified as 5 W and if the profit (benefit B) associated with the item which caused the health issue (e.g. contaminated meat) is 0.1 W, then D is calculated as 50 B.

Finally, we also introduce the following notation:

N	Number of items in the lot
M	Number of nonconforming items in the lot
n	Sample size (i.e. number of items in the sample)

The utility function is defined as follows:

$$\mathrm{U}(N, B, M, D, T, n) = \begin{cases} B \cdot N - D \cdot M - T \cdot n, & \text{if the lot is accepted} \\ -T \cdot n, & \text{if the lot is rejected} \end{cases}$$

In the case of an accepted lot, the utility function can be rewritten as

$$\mathrm{U}(N, B, M, D, T, n) = N \cdot \left(B - D \cdot \frac{M}{N} - T \cdot \frac{n}{N}\right)$$

It should be noted that M is an integer which, in general, remains unknown. The aim of the acceptance sampling procedure is to obtain an estimate of U by means of a suitable (nonbiased) estimator for M (or for the proportion of nonconforming items $\frac{M}{N}$), taking all prior information into account.

6.3.4 Example

In order to illustrate the concept of utility as well as the coefficients for the benefit and damages discussed in the previous section, we consider the case where the utility is simply a given value rather than the expected value of a random variable.

Consider the scenario where a retailer has accepted and purchased a lot of 2000 apples (400 kg) at a wholesale price of 600 €. The retailer expects to sell all apples at an average retail price of 4 € per kg. Total sales (if all apples are sold) for the lot will thus be 1600 €. Transport costs were 20 €. The retailer paid a salesperson 100 € for the day's work.

The benefit for the entire lot (under the assumption that all the apples are sold) for the retailer is thus

$$1600\,€ - 600\,€ - 20\,€ - 100\,€ = 880\,€^{8}$$

A total of 100 apples (20 kg) had blemishes which resulted in being discarded (i.e. not sold). The retailer concludes that the damages caused by nonconforming items are 80 €.

[8] For the sake of simplicity, neither overhead costs (associated with the retail outlet) nor taxes are included here.

Benefit, damages and testing costs for the example

Utility for a lot which has been **accepted**	=	benefit associated with an accepted lot (under the assumption that all items are conforming) i.e. returns minus expenditures	880 €
		minus damages associated with nonconforming items in an accepted lot	**minus** 80 €.
		minus sampling and testing costs	**minus** 0 €.

Accordingly, in this example, the utility for the lot is **800 €.** Now, let us calculate the parameters B and D (we already know that $T = 0$). The benefit per conforming item (apple without blemishes) B expressed in Euros is

$$B = \frac{880\ €}{2000 \text{ apples in the lot}} = 0.44\ € \text{ per apple}$$

The damages associated with nonconforming items are

$$D = \frac{80\ €}{100 \text{ apples with blemishes}} = 0.8\ € \text{ per apple}$$

If the damages coefficient is expressed in terms of *B*, we have

$$D = \frac{0.8\ € \text{ per apple}}{0.44\ € \text{ per apple}} = 1.82\ B \text{ per apple}$$

The following table summarizes the example.

Table 8: Summary of example of lot of 1000 apples

Lot size	$N = 2000$
Sample size	$n = 0$
Acceptance number	c Not applicable
Sampling and testing costs	$T = 0\ B$
Damages per nonconforming item	$D = 1.82\ B$
Conversion of benefit per conforming item	$B = 0.44\ €$
Proportion of nonconforming items	x_0 = 5 %
Utility	$B \cdot N - D \cdot M - T \cdot n$ $= 0.44\ € \cdot 2000 - 1.82 \cdot 0.88\ € \cdot 100$ $= 800\ €$

Note 1

If the retailer purchases lemons instead of apples and if the lemons are displayed for purchase at the retail outlet during 10 days, then the retailer will worry about mold spread via contamination with neighboring spoilt lemons. If the lemons are sold individually and if the retailer observes that, on average, one spoilt lemon contaminates 5 neighboring lemons in the time span of 10 days, this state of affairs can be taken into consideration by multiplying the D coefficient by 5.

Note 2

If an intermediate distributor is involved in the purchase of a lot of 500 crates of 2000 apples (10^6 apples), it is likely that the lot will be inspected prior to acceptance. In such a situation, T will be nonzero.

Note 3

If the aim is to use the utility in order to determine the acceptance sampling plan (sample size n and acceptance number c), then the damages coefficient can be specified via the mean value of the prior distribution for the percentage nonconforming.

In order to illustrate this point, we start with the observation that, in the example above, at least 900 apples must be sold in order for the retailer to break even:

Expenditures (Purchase price of the lot + transport costs + sales person remuneration)	720 €
Retail price of apple	0.8 €
Income from the sale of 900 apples (180 kg)	720 €

Accordingly, 1 maximum of 1100 nonconforming items (1100 apples with blemishes leading to discarding) can be tolerated in order to break even. This corresponds to a maximum of 55% for the percentage nonconforming.

If the retailer works with a prior distribution for the percentage nonconforming whose mean value is 10% (i.e. well below the maximum of 55%), then *D* can be determined as follows. First, we note that the percentage nonconforming corresponds to $\frac{M}{N}$. In the absence of testing costs, the utility is simply

$$U = N \cdot B - D \cdot M$$

Setting $U = 0$ (i.e. lot acceptance in the sense of breaking even), we obtain the following expression for the damages coefficient

$$D = \frac{N}{M} \cdot B$$

If D is expressed in terms of B, then this simplifies to

$$D = \frac{N}{M}$$

For $\frac{M}{N} = 0.1$ (the prior which the retailer is working with), we thus obtain $D = 10$.

Note 4

It is important to understand the impact which D has on the utility. In particular, in the absence of sampling and testing costs, the value $D = 1\ B$ means that one single sold item is sufficient to have a positive utility.

Indeed, for $M = N - 1$ and $D = 1\ B$ we have

$$U = N \cdot B - D \cdot M = N - M = N - (N - 1) = 1$$

6.3.5 The role of the prior

In the example from the previous section, the lot has already been accepted and purchased and the proportion of nonconforming items x_0 can be empirically determined by 100% inspection of the lot. In other words, in such a case, x_0 is an empirically determined value rather than a random variable (hence the notation with the subscript). However, the aim of the utility approach is to determine sample size and acceptance number—in other words to design the acceptance sampling plan—prior to lot inspection. For this reason, in the following, the empirically determined x_0 value will be replaced with a random variable X whose distribution as called the *prior*. This gives a slightly different meaning to the utility: instead of an *empirical* value corresponding to a given lot, it is now an *expected* value, in the probabilistic sense. The expected utility for a given plan (sample size n and acceptance number c) calculated on the basis of the prior distribution is denoted $u_{prior}(n, c)$.

6.3.6 Utility curves

The following diagrams show utility curves:

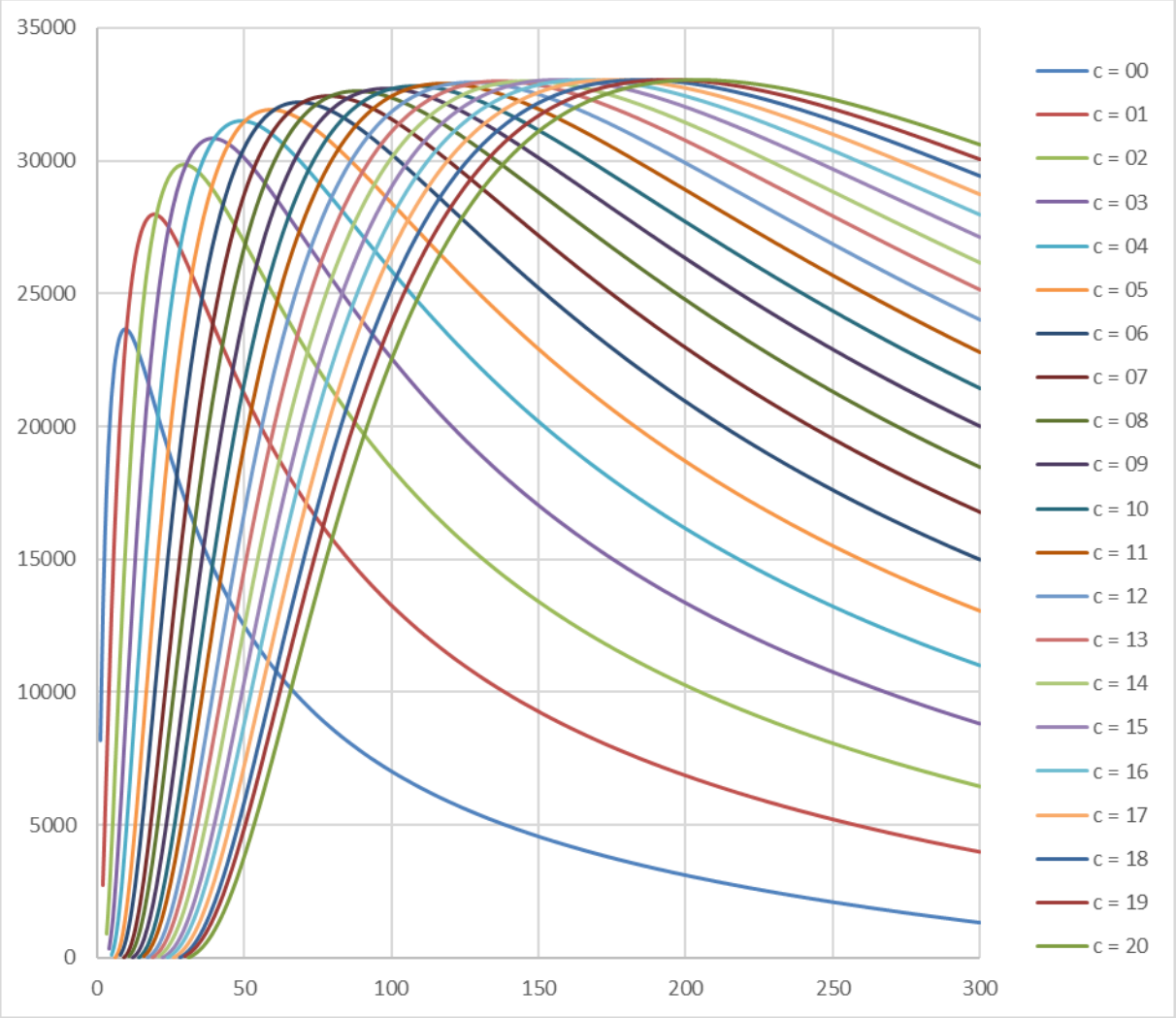
- for the prior Beta(1,9) with mean value 10% percentage nonconforming
- for the prior Beta(0.5,0.5) with mean value 50% percentage nonconforming (“non-informative” prior)

The x-axis shows the sample size n and the y-axis shows the expected utility $u_{prior}(n, c)$. There is a separate curve for each acceptance number c.

As can be seen, the utility values for the more optimistic prior are greater (maximum around 33000 B) than for the non-informative prior (maximum around 12600 B).

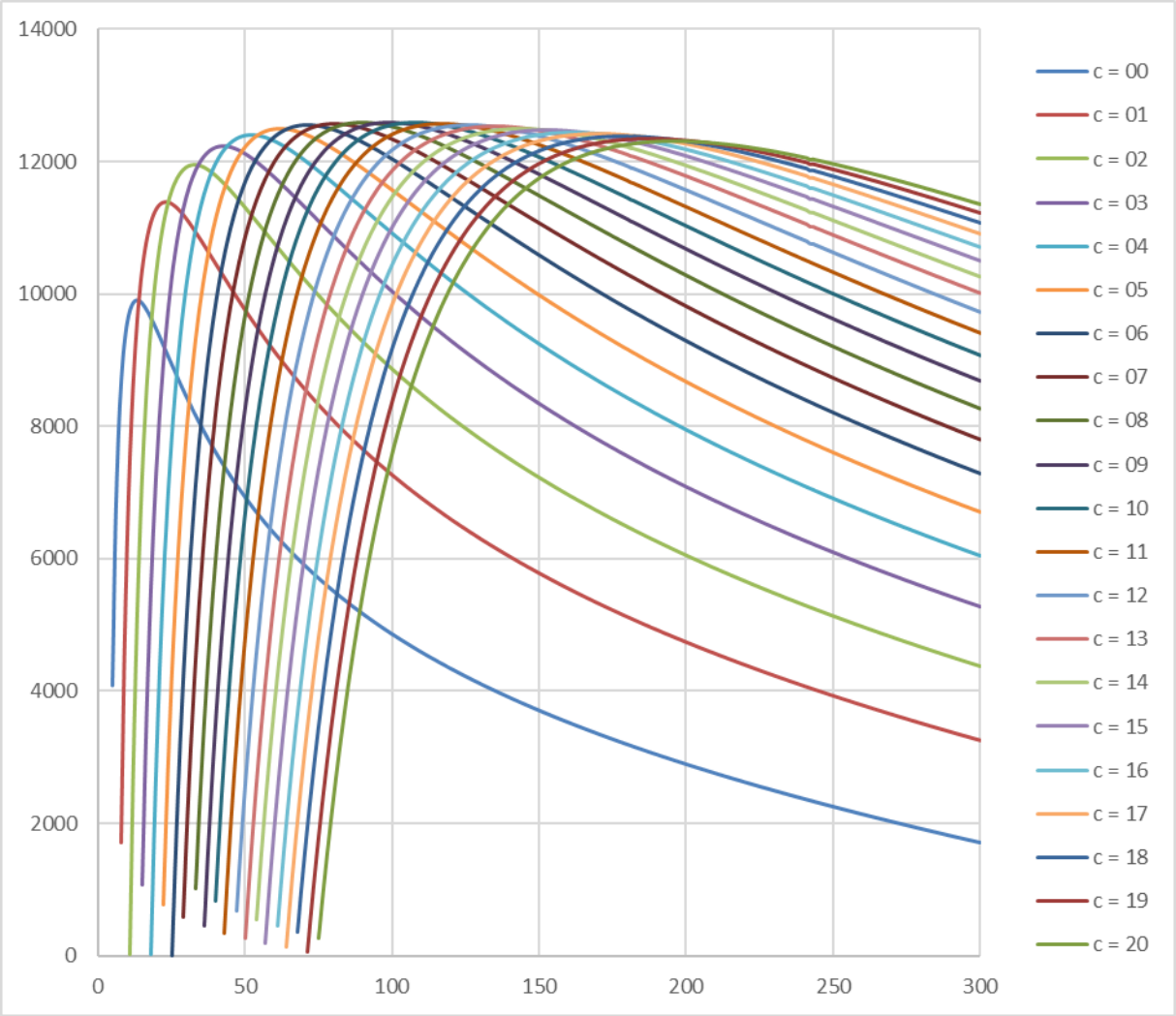
The following diagram shows utility curves for the prior Beta(1,9), lot size N = 100000 and cost structure $D = 10\ B$ and $T = 5\ B$. The optimum plan is $n = 175$, $c = 17$ (utility = 33043 B).

Figure 41: Utility curves for different acceptance number c values as a function of sample size n for the prior Beta(1,9), lot size N = 100000 and the cost structure D = 10 B and T = 5 B



The following diagram shows the utility curves for the Jeffreys prior Beta(0.5,0.5), which represents the case that no prior information is available (noninformative prior). As in the previous diagram, the lot size N is 100000 and the cost structure is $D = 10\ B$ and $T = 5\ B$. The optimum plan is $n = 99$, $c = 9$ (utility = 12592 B).

Figure 42: Utility curves for different acceptance number c values as a function of sample size n for the prior Beta(0.5,0.5), lot size N = 100000 and the cost structure D = 10 B and T = 5 B



As can be seen in the above diagrams, the utility curves appear to plateau around the maximum value. This motivates the following procedure: we propose to consider plans whose utility lies within 10% of the maximum utility. This will allow a considerable reduction in sample size while maintaining a negligible impact on utility.

For instance, the maximum utility for the Beta(1,9) prior is 30457 B. Subtracting 10% of this maximum value, we obtain 29739 B. The $c = 2$ curve exceeds this value at $n = 27$. Hence the $n = 175$, $c = 17$ plan can be replaced with $n = 27$, $c = 2$.

6.3.7 Standard plans

This section provides tables with standard plans for the practitioner. It is assumed that there is no inspection error and that prior information is available in the form of results from previous testing performed on items from the producer of the lot currently under inspection. It is assumed that these previous tests have been performed recently—say, within the previous 2 or 3 months. In the tables, the results from previous testing are represented via the pair (n_0, y_0) with n_0 denoting the number of items previously tested and y_0 denoting the number of nonconforming items from these recent tests.

For a given pair (n_0, y_0), the plans are calculated on the basis of a Beta(α, β) prior for the percentage nonconforming where

$$\alpha = y_0 + 0.5$$

$$\beta = n_0 - y_0 + 0.5$$

The pair $(n_0 = 0, y_0 = 0)$ stands for the absence of any prior information. This scenario is represented as the first row in each of the tables. The corresponding standard plans are based on the noninformative Jeffreys prior, i.e. the Beta(0.5,0.5) distribution.

The standard plans can be used to answer two questions.

Question 1: Given prior information, a cost structure and a lot size, which acceptance sampling plan should be applied?

Question 2: Given a cost structure and lot size, what type of prior information is required in order to achieve an acceptance sampling plan whose workload is acceptable (or indeed, for it to be worthwhile to perform acceptance sampling at all)

In order to illustrate the use of the standard plans to answer these two questions, consider the following examples.

Example 1
A lot with size $N = 100\,000$ packages of grains was purchased at a price of 100 000 GCU (where GCU stands for generic currency unit). The retailer who purchased the lot intends to sell each package at 6 GCU. A simplified calculation (i.e. without taking overhead costs etc. into consideration) thus yields a benefit per item of $B = 5$ GCU. Say that sampling and testing costs are 25 GCU per package. The corresponding parameter is thus $T = 5\,B$. Finally, if the damages associated with a nonconforming item consist in a dissatisfied customer who may no longer return to the retailer's outlet, the D parameter can be specified as $D = 10\,B$.
Answer to question 1: Given this background, if the retailer has recently purchased another lot and tested 20 packages, none of which were nonconforming, then the plan $n = 33$, $c = 1$ can be applied.
Answer to question 2: If the retailer would like to test, say, no more than 10 packages, then prior information based on at least 50 tested packages would be required.
Example 2
A lot of canned salmon is inspected following the item conformity criteria set out in Section 8 of CXS 3-1981. Say the lot size is $N = 1000$ items (cans) and that the sampling and testing costs per item are $T = 5\,B$ (as in Example 1). Finally, the damages associated with a nonconforming item consist in a dissatisfied customer who may no longer to the retailer's outlet, and the D parameter can be specified as $D = 10\,B$ (as in Example 1).
Answer to question 1: Given this background, if the consumer has data from 5 recently tested cans from the same supplier, one of which was nonconforming, the decision would be to reject without testing.
Answer to question 2: The consumer would need to test an additional three cans prior to investing resources in inspecting a lot of 1000 cans. With prior information $n_0 = 8$, $y_0 = 1$, the acceptance sampling plan would be (10,0).

Notation

The following notation is used in the tables:

n_0	Previous testing / prior information The number of items tested prior to the current lot inspection
y_0	Previous testing / prior information The number of nonconforming items
N	Lot size
D	Damages/losses/costs per nonconforming item (expressed in terms of the benefit per item B)
T	Sampling and testing costs per item (expressed in terms of the benefit per item B)
a	Accept without testing
r	Reject without testing
(n, c)	Acceptance sampling plan with sample size n acceptance number c

Table 9: Standard plans for testing and sampling costs $T = 5\ B$

Prior test results		N	1000					10000					100000				
		D	1.5	3	10	30	100	1.5	3	10	30	100	1.5	3	10	30	100
		T	5					5					5				
n_0	y_0	T/N	0.500%					0.050%					0.005%				
0	0		(1,0)	(2,0)	(7,0)	r	r	(2,1)	(5,1)	(15,1)	(25,0)	r	(2,1)	(7,2)	(21,1)	(50,1)	(93,0)
1	0		a	(2,0)	(7,0)	r	r	a	(4,1)	(15,1)	(27,0)	(78,0)	a	(4,1)	(24,2)	(53,1)	(110,0)
2	0		a	(1,0)	(6,0)	r	r	a	(3,1)	(14,1)	(28,0)	(77,0)	a	(3,1)	(20,1)	(54,1)	(119,0)
3	0		a	(1,0)	(5,0)	(20,0)	r	a	(1,0)	(13,1)	(28,0)	(76,0)	a	(3,1)	(18,1)	(54,1)	(131,0)
4	0		a	(1,0)	(5,0)	(19,0)	r	a	(1,0)	(12,1)	(28,0)	(76,0)	a	(1,0)	(16,1)	(53,1)	(147,1)
5	0		a	(1,0)	(4,0)	(18,0)	r	a	(1,0)	(11,1)	(28,0)	(76,0)	a	(1,0)	(14,1)	(52,1)	(147,1)
8	0		a	(1,0)	(3,0)	(15,0)	r	a	(1,0)	(7,0)	(26,0)	(74,0)	a	(1,0)	(10,1)	(49,1)	(144,1)
10	0		a	(1,0)	(3,0)	(14,0)	r	a	(1,0)	(5,0)	(24,0)	(73,0)	a	(1,0)	(9,1)	(46,1)	(143,1)
13	0		a	(1,0)	(2,0)	(12,0)	r	a	(1,0)	(4,0)	(21,0)	(71,0)	a	(1,0)	(7,1)	(42,1)	(140,1)
20	0		a	(1,0)	(2,0)	(8,0)	r	a	(1,0)	(3,0)	(15,0)	(65,0)	a	(1,0)	(3,0)	(33,1)	(133,1)
30	0		a	(1,0)	(2,0)	(6,0)	r	a	(1,0)	(3,0)	(9,0)	(57,0)	a	(1,0)	(3,0)	(22,1)	(122,1)
50	0		a	(1,0)	(2,0)	(5,0)	(22,0)	a	(1,0)	(2,0)	(7,0)	(41,0)	a	(1,0)	(3,0)	(9,0)	(80,0)
80	0		a	(1,0)	(2,0)	(4,0)	(14,0)	a	(1,0)	(2,0)	(6,0)	(23,0)	a	(1,0)	(3,0)	(7,0)	(50,0)
100	0		a	(1,0)	(2,0)	(4,0)	(12,0)	a	(1,0)	(2,0)	(6,0)	(18,0)	a	(1,0)	(3,0)	(7,0)	(34,0)
1	1		(2,1)	(3,0)	r	r	r	(4,2)	(7,1)	(11,0)	r	r	(6,3)	(12,3)	(22,1)	(36,0)	(87,0)
2	1		a	(5,1)	r	r	r	(2,1)	(9,2)	(16,0)	r	r	(3,2)	(15,4)	(33,2)	(59,1)	r
3	1		a	(4,1)	r	r	r	a	(8,2)	(21,1)	r	r	a	(14,4)	(41,3)	(64,1)	r
4	1		a	(3,1)	r	r	r	a	(7,2)	(22,1)	r	r	a	(10,3)	(43,3)	(69,1)	r
5	1		a	(3,1)	r	r	r	a	(6,2)	(22,1)	r	r	a	(8,3)	(46,3)	(72,1)	r
8	1		a	(1,0)	(10,0)	r	r	a	(2,1)	(23,1)	r	r	a	(3,1)	(48,4)	(96,2)	r
10	1		a	(1,0)	(8,0)	r	r	a	(2,1)	(25,2)	(45,0)	r	a	(2,1)	(45,4)	(98,2)	r
13	1		a	(1,0)	(6,0)	r	r	a	(1,0)	(19,1)	(42,0)	r	a	(2,1)	(37,3)	(102,2)	(159,0)
20	1		a	(1,0)	(3,0)	r	r	a	(1,0)	(10,1)	(58,1)	r	a	(1,0)	(20,2)	(106,2)	(159,0)
30	1		a	(1,0)	(3,0)	r	r	a	(1,0)	(6,1)	(49,1)	r	a	(1,0)	(7,1)	(97,2)	(156,0)
50	1		a	(1,0)	(2,0)	(9,0)	r	a	(1,0)	(4,0)	(30,1)	r	a	(1,0)	(5,1)	(64,2)	(147,0)
80	1		a	(1,0)	(2,0)	(6,0)	r	a	(1,0)	(3,0)	(10,0)	r	a	(1,0)	(3,0)	(20,1)	(180,1)
100	1		a	(1,0)	(2,0)	(5,0)	r	a	(1,0)	(3,0)	(8,0)	(87,0)	a	(1,0)	(3,0)	(15,1)	(167,1)
8	2		a	(3,1)	r	r	r	a	(6,2)	(25,1)	r	r	a	(12,4)	(48,3)	(68,1)	r
10	2		a	(1,0)	r	r	r	a	(3,1)	(25,1)	r	r	a	(6,2)	(56,4)	(74,1)	r
13	2		a	(1,0)	r	r	r	a	(2,1)	(26,1)	r	r	a	(3,1)	(62,5)	(80,1)	r
20	2		a	(1,0)	(8,0)	r	r	a	(2,1)	(26,2)	r	r	a	(2,1)	(56,5)	(109,2)	r
30	2		a	(1,0)	(3,0)	r	r	a	(1,0)	(12,1)	r	r	a	(2,1)	(30,3)	(133,3)	r
50	2		a	(1,0)	(3,0)	r	r	a	(1,0)	(6,1)	(59,1)	r	a	(1,0)	(7,1)	(122,3)	r

Prior test results		N	1000					10000					100000				
		D	1.5	3	10	30	100	1.5	3	10	30	100	1.5	3	10	30	100
		T	5					5					5				
n_0	y_0	T/N	0.500%					0.050%					0.005%				
80	2		a	(1,0)	(2,0)	(10,0)	r	a	(1,0)	(5,1)	(32,1)	r	a	(1,0)	(6,1)	(87,3)	r
100	2		a	(1,0)	(2,0)	(7,0)	r	a	(1,0)	(3,0)	(19,1)	r	a	(1,0)	(5,1)	(46,2)	(186,0)
20	3		a	(1,0)	r	r	r	a	(2,1)	(27,1)	r	r	a	(2,1)	(70,5)	(81,1)	r
30	3		a	(1,0)	(8,0)	r	r	a	(2,1)	(27,2)	r	r	a	(2,1)	(66,6)	(92,1)	r
50	3		a	(1,0)	(3,0)	r	r	a	(1,0)	(7,1)	r	r	a	(1,0)	(15,2)	(143,3)	r
80	3		a	(1,0)	(2,0)	r	r	a	(1,0)	(5,1)	(60,1)	r	a	(1,0)	(6,1)	(127,3)	r
100	3		a	(1,0)	(2,0)	r	r	a	(1,0)	(5,1)	(41,1)	r	a	(1,0)	(6,1)	(107,3)	r
50	4		a	(1,0)	(4,0)	r	r	a	(1,0)	(17,2)	r	r	a	(2,1)	(46,5)	(100,1)	r
80	4		a	(1,0)	(3,0)	r	r	a	(1,0)	(6,1)	r	r	a	(1,0)	(8,1)	(147,3)	r
100	4		a	(1,0)	(3,0)	r	r	a	(1,0)	(5,1)	r	r	a	(1,0)	(6,1)	(142,3)	r

Table 10: Standard plans for testing and sampling costs $T = 25\ B$

Prior test results		N	1000					10000					100000				
		D	1.5	3	10	30	100	1.5	3	10	30	100	1.5	3	10	30	100
		T	25					25					25				
n_0	y_0	T/N	2.500%					0.250%					0.025%				
0	0		(1,0)	(2,0)	r	r	r	(2,1)	(4,1)	(8,0)	r	r	(2,1)	(5,1)	(16,1)	(29,0)	(78,0)
1	0		a	(1,0)	r	r	r	a	(2,0)	(8,0)	(22,0)	r	a	(4,1)	(16,1)	(34,0)	(80,0)
2	0		a	(1,0)	(5,0)	r	r	a	(1,0)	(7,0)	(21,0)	r	a	(3,1)	(15,1)	(44,1)	(81,0)
3	0		a	(1,0)	(4,0)	r	r	a	(1,0)	(7,0)	(21,0)	r	a	(1,0)	(14,1)	(43,1)	(81,0)
4	0		a	(1,0)	(4,0)	r	r	a	(1,0)	(6,0)	(20,0)	r	a	(1,0)	(13,1)	(43,1)	(81,0)
5	0		a	(1,0)	(3,0)	r	r	a	(1,0)	(5,0)	(19,0)	r	a	(1,0)	(12,1)	(42,1)	(81,0)
8	0		a	(1,0)	(2,0)	r	r	a	(1,0)	(4,0)	(17,0)	r	a	(1,0)	(9,1)	(39,1)	(80,0)
10	0		a	(1,0)	(2,0)	r	r	a	(1,0)	(3,0)	(15,0)	r	a	(1,0)	(7,1)	(37,1)	(80,0)
13	0		a	(1,0)	(2,0)	r	r	a	(1,0)	(3,0)	(13,0)	r	a	(1,0)	(4,0)	(34,1)	(80,0)
20	0		a	(1,0)	(2,0)	(5,0)	r	a	(1,0)	(2,0)	(9,0)	(55,0)	a	(1,0)	(3,0)	(22,0)	(77,0)
30	0		a	(1,0)	(2,0)	(4,0)	r	a	(1,0)	(2,0)	(6,0)	(44,0)	a	(1,0)	(3,0)	(11,0)	(70,0)
50	0		a	(1,0)	(2,0)	(4,0)	r	a	(1,0)	(2,0)	(5,0)	(28,0)	a	(1,0)	(3,0)	(7,0)	(53,0)
80	0		a	(1,0)	(2,0)	(3,0)	r	a	(1,0)	(2,0)	(5,0)	(16,0)	a	(1,0)	(2,0)	(6,0)	(30,0)
100	0		a	(1,0)	(2,0)	(3,0)	(9,0)	a	(1,0)	(2,0)	(5,0)	(14,0)	a	(1,0)	(2,0)	(6,0)	(22,0)
1	1		(1,0)	r	r	r	r	(2,1)	(4,0)	r	r	r	(4,2)	(9,2)	(15,0)	(29,0)	r
2	1		a	(2,0)	r	r	r	(2,1)	(5,1)	r	r	r	(2,1)	(11,3)	(22,1)	(36,0)	r
3	1		a	(2,0)	r	r	r	a	(5,1)	r	r	r	a	(10,3)	(24,1)	(40,0)	r
4	1		a	(1,0)	r	r	r	a	(4,1)	(13,0)	r	r	a	(8,2)	(30,2)	(42,0)	r
5	1		a	(1,0)	r	r	r	a	(3,1)	(12,0)	r	r	a	(6,2)	(30,2)	(43,0)	r
8	1		a	(1,0)	r	r	r	a	(1,0)	(11,0)	r	r	a	(2,1)	(29,2)	(45,0)	r
10	1		a	(1,0)	r	r	r	a	(1,0)	(10,0)	r	r	a	(2,1)	(28,2)	(66,1)	r
13	1		a	(1,0)	r	r	r	a	(1,0)	(9,0)	r	r	a	(2,1)	(24,2)	(65,1)	r
20	1		a	(1,0)	(2,0)	r	r	a	(1,0)	(4,0)	r	r	a	(1,0)	(12,1)	(63,1)	r
30	1		a	(1,0)	(2,0)	r	r	a	(1,0)	(3,0)	(25,0)	r	a	(1,0)	(6,1)	(58,1)	r
50	1		a	(1,0)	(2,0)	r	r	a	(1,0)	(2,0)	(11,0)	r	a	(1,0)	(5,1)	(37,1)	r
80	1		a	(1,0)	(2,0)	(4,0)	r	a	(1,0)	(2,0)	(6,0)	r	a	(1,0)	(3,0)	(16,1)	(111,0)
100	1		a	(1,0)	(2,0)	(4,0)	r	a	(1,0)	(2,0)	(6,0)	r	a	(1,0)	(3,0)	(11,0)	(111,0)
8	2		a	(1,0)	r	r	r	a	(3,1)	r	r	r	a	(9,3)	(27,1)	r	r
10	2		a	(1,0)	r	r	r	a	(2,1)	r	r	r	a	(5,2)	(34,2)	r	r
13	2		a	(1,0)	r	r	r	a	(2,1)	r	r	r	a	(2,1)	(35,2)	r	r
20	2		a	(1,0)	r	r	r	a	(1,0)	(9,0)	r	r	a	(2,1)	(35,3)	r	r
30	2		a	(1,0)	(3,0)	r	r	a	(1,0)	(5,0)	r	r	a	(1,0)	(18,2)	(78,1)	r

Prior test results		N	1000					10000					100000				
		D	1.5	3	10	30	100	1.5	3	10	30	100	1.5	3	10	30	100
		T	25					25					25				
n_0	y_0	T/N	2.500%					0.250%					0.025%				
50	2		a	(1,0)	(2,0)	r	r	a	(1,0)	(3,0)	r	r	a	(1,0)	(6,1)	(68,1)	r
80	2		a	(1,0)	(2,0)	r	r	a	(1,0)	(3,0)	(11,0)	r	a	(1,0)	(5,1)	(41,1)	r
100	2		a	(1,0)	(2,0)	(5,0)	r	a	(1,0)	(2,0)	(7,0)	r	a	(1,0)	(5,1)	(22,1)	r
20	3		a	(1,0)	r	r	r	a	(2,1)	r	r	r	a	(2,1)	(37,2)	r	r
30	3		a	(1,0)	r	r	r	a	(1,0)	(9,0)	r	r	a	(2,1)	(37,3)	r	r
50	3		a	a	(2,0)	r	r	a	(1,0)	(4,0)	r	r	a	(1,0)	(9,1)	r	r
80	3		a	(1,0)	(2,0)	r	r	a	(1,0)	(3,0)	r	r	a	(1,0)	(6,1)	(69,1)	r
100	3		a	(1,0)	(2,0)	r	r	a	(1,0)	(3,0)	(16,0)	r	a	(1,0)	(5,1)	(55,1)	r
50	4		a	(1,0)	(3,0)	r	r	a	(1,0)	(5,0)	r	r	a	(2,1)	(21,2)	r	r
80	4		a	(1,0)	(2,0)	r	r	a	(1,0)	(5,1)	r	r	a	(1,0)	(6,1)	r	r
100	4		a	(1,0)	(2,0)	r	r	a	(1,0)	(3,0)	r	r	a	(1,0)	(6,1)	(73,1)	r

General notes

Note 1

As the lot size N increases, the sample size also increases, but at the same time, it becomes “easier” to justify investing resources in lot inspection—i.e. plans are available at lower n_0 values. This is due to the increase in benefit due to the increased lot size.

Note 2

For a given cost structure and lot size, the sample size is less for $T = 25\ B$ than for $T = 5\ B$. On the other hand, a higher n_0 value is required prior to committing resources to acceptance sampling for $T = 25\ B$.

Note 3

The ‘r’ entries (highlighted in red in the table) indicate that the lot should be rejected without testing. This should be interpreted as follows: given the testing costs and the damages associated with nonconforming items, it only makes sense to invest resources in lot inspection given a minimum level of confidence in the lot quality. This minimum level of confidence is codified via the (n_0, y_0) pair. For example, for a relatively small lot ($N = 1000$) and a very high value for D ($D = 100$, reflecting for example a health hazard in connection with nonconforming items), it only makes sense to conduct acceptance sampling if the prior information is based on at least 50 previously tested items with zero nonconforming results.

If the consumer rejects the lot without testing, the producer has the following options:

1. Increase the lot size N. As seen in the standard plans, a larger lot size translates to higher income for the consumer, thus lowering the threshold for investing resources in acceptance sampling.
2. Decrease the purchase price of the lot. This is tantamount to increasing the parameter B, which, in turn, will result in lower values for D and T. This will lower the threshold for investing resources in acceptance sampling.
3. Develop test methods which are cheaper to apply. This option will result in a decrease in the T parameter, thus lowering the threshold for investing resources in acceptance sampling. An important caveat here is that the performance of the new method must be at least as good as that of the original method.

Technical notes

Note 1

The standard plans were calculated via a hierarchical Bayesian model which “mixes” two priors: the “actual” prior corresponding to the prior information and the “non-informative” Jeffreys prior. The latter’s influence is increased the further the testing outcome deviates from the “actual” prior. See Uhlig et al. (2025) [25]. In addition, the 10% approximation (see discussion at the end of the previous section) is applied.

Note 2

We define the ratio $x_0 = B/D$ (or $1/D$ if D is expressed in terms of B). The probability that the percentage nonconforming exceeds x_0 is an interesting pendant to the consumer’s risk in the ISO 2859 series of standards. See the discussion in Hald [23].

Note 3

The sample size increases with n_0 as long as the mean value of the beta distribution corresponding to (n_0, y_0) is less than the ratio $x_0 = B/D$ (or $1/D$ if D is expressed in terms of B). When this mean value is greater than x_0, the sample size decreases as n_0 increases. This can be explained as follows: if the mean value is less than x_0, it is unlikely that utility will be positive, and the natural tendency of the model is to resist investing resources in lot inspection. This resistance decreases as the prior becomes more optimistic. By contrast, if the mean value is greater than x_0, then it is likely that the utility will be positive, and the natural tendency of the model is to invest resources in lot inspection. As the prior is becomes more optimistic, fewer resources are required.

Note 4

The standard plan for $N = 1000$, $D = 3$, $T = 25$, $n_0 = 50$, $y_0 = 3$ is “accept” rather than (1,0). This anomaly may possibly be related to rounding issues.

More information regarding this approach can also be found in Uhlig et al. (2025) [25].

6.3.8 Broader view of utility

As can be seen in the standard plans, the acceptance sampling plan depends very much on the lot size. Indeed, a large lot size translates to an increase in total benefit, thus impacting the calculation of utility. For example, for the cost structure $D = 30\,B$ and $T = 25\,B$, and for the prior information $(n_0 = 20, y_0 = 1)$, there is an acceptance plan (i.e. it makes sense to invest resources in lot inspection) only for the lot size $N = 100\,000$. Indeed, for the lot size $N = 1000$ and the lot size $N = 10\,000$, the utility approach results in the decision to reject the lot without testing. These considerations show the extent to which the acceptance samplings plans (including the decision to reject without testing) reflect the cost structure internalized in the parameters of the utility model.

There are three takeaways from this for the consumer.

The first is that the consumer can perform some preliminary calculations and inform the producer prior to the lot being shipped that, given the cost structure, the transaction is only commercially viable for a minimum lot size.

The second is that, in certain circumstances, it could lie in the interest of the consumer to “pretend” that the lot size is greater than it actually is in order to achieve a viable acceptance sampling plan. For instance, consider the case that an importing country is initiating commercial relations with a new supplier and that the first lot—intended as a trial—is smaller in size than subsequent “routine” lots would be.

The third and last takeaway is that an indispensable condition for achieving plans which successfully balance the producer’s and the consumer’s interests is transparency. For instance, the producer must be able to ascertain whether the consumer intends to reject without testing or to apply an acceptance sampling plan *prior to shipping the lot*. Indeed, this last take away leads directly to the question whether it is possible to combine utility functions representing the consumer’s and the producer’s perspectives so as to achieve a broader notion of utility representing, as it were, a win-win situation for both parties to the transaction. This is discussed in the following section.

6.3.9 Adversarial approach

The standard plans from the previous section were calculated on the basis of a utility concept which reflects the consumer’s perspective. However, the utility approach can be extended to include the producer’s perspective. This will be briefly described in this section.

If the consumer reaches the decision to reject without testing, then the producer has two possible courses of action.

1. Apply one of the options listed under **Note 3** in the **General notes,** at the end of Section 6.3.7
2. Offer to pay for testing and sampling costs. In such a case, the sampling plan can be calculated from the point of view of the producer. This is where the *adversarial* approach is applied. The aim of the adversarial approach is to calculate an acceptance sampling plan (sample size and acceptance number) by maximizing the producer’s utility. The latter in turn, takes into account the consumer’s decision to accept or reject the lot. In other words, the adversarial approach consists in combining the consumer’s and the producer’s utility functions.

The adversarial approach was first described in articles by Lindley and Singpurwalla [24]. For further information regarding the adversarial approach, the reader is referred to Uhlig et al. (2025) [26].

6.4 Bayesian plans: glossary of terms

Percentage nonconforming	The proportion of nonconforming items in the lot, expressed as a percentage. In the ISO 2859 and ISO 3951 standards, this is called percentage nonconforming.
Prior distribution (short: prior)	Statistical distribution which encapsulates information regarding the lot quality which is available prior to the lot inspection
Posterior distribution (short: posterior)	Statistical distribution which combines the prior and the testing outcomes
Hyperparameter	Parameter of the prior or posterior distribution
Beta distribution	Typical choice of prior for the percentage nonconforming in the case of inspection by attributes
Parametric risk ("classical" or "ISO" risk)	The producer's and consumer's risks as defined in the ISO 2859 and ISO 3951 standards. These risks start from a given quality level and calculate the corresponding probability of acceptance or rejection. In other words, the quality level is treated as the parameter of a statistical distribution.
Bayesian risk	These risks are calculated via a prior distribution for the parameter which characterizes the lot quality. For example, in the case of inspection by attributes, the risks are calculated via a prior distribution for the percentage nonconforming. See Section 6.2.
Lot conformity	In the case of inspection by attributes: a lot is conforming if the percentage nonconforming lies within a conformance region $\mathcal{C}$ for lot quality specified via an upper limit $x_{\mathcal{C}}$ for the percentage nonconforming (e.g. upper limit $x_{\mathcal{C}} = 10\%$). Lot conformity must be carefully distinguished from item conformity.
Conformance probability	The probability that a lot is conforming, given a test outcome.
Specific consumer risk	A type of Bayesian risk. The probability that a lot is nonconforming given a test outcome resulting in lot acceptance.
Global producer risk	A type of Bayesian risk. The probability that lot is both conforming and rejected.
Utility	A value reflecting benefits and costs associated with an acceptance sampling plan and a lot.
Expected prior utility	Insofar as the utility is calculated from a prior for the percentage nonconforming, the utility is a random variable. The expected value of the utility is called the expected prior utility.

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APPENDIX V

NUMERIC PERFORMANCE CRITERIA FOR NITRATE AND NITRITE IONS IN CERTAIN FOOD MATRICES

(For CCFA's consideration)

Table 1: Numeric Performance Criteria for adopted MLs

Food Additive	Subcategory for which value was provided	Adopted Maximum Levels (CXS 192-1995)*	Calculated method performance criteria based on Maximum level (mg/kg)					
			Min Appl. Range (mg/kg)	LOD (mg/kg)	LOQ (mg/kg)	Precision (RSD_R (%))	Recovery (%)	Examples of applicable methods that meet the criteria
01.6 (*Cheese and analogues)*								
Nitrate	01.6.2 *(Ripened cheese)*	35 mg/kg as residual NO_3 ion.	25 – 45	3.5	7	19	80 – 110	**Multi-laboratory validation** - ISO 14673-3 I IDF 189-3: 2004 **Single-laboratory validation -** ISO 14673-2 I IDF 189-2: 2004^
08.0 (*Meat and meat products, including poultry and game)*								
Nitrite	08.2.2 (Heat-treated processed meat, poultry, and game products in whole pieces or cuts/)	80 mg/kg as residual NO_2 ion.	60 – 110	8	16	17	80 – 110	**Multi-laboratory validation** - AOAC Method 973.31; NMKL 165: 2000 Ed.; **Single-laboratory validation** - Afanda et al., (2025); Iammarino et al. 2013; Ferreira et al. (2008) for Ham; Siu et al., 1998 for Salami and Ham
Nitrite	08.3 (Processed comminuted meat, poultry, and game products)	80 mg/kg as residual NO_2 ion.	60 – 110	8	16	17	80 – 110	**Multi-laboratory validation** - AOAC Method 973.31; NMKL 165: 2000 Ed.; **Single-laboratory validation** - Afanda et al., (2025); Iammarino et al., 2013; Ferreira et al., (2008) for Ham; Siu et al., 1998 for Salami and Ham

Notes: * Maximum levels in CXS 192-1995.

^ In the absence of LOD or LOQ being specified in the method, the collaborative study report being unavailable at-this-time, and relying on in-house validation data, the validation designated as SLV, although the SLV status may be reviewed with additional data.

Table 2: Numeric performance criteria for the lowest proposed residual levels for representative provisions in dairy (cheese), meat, and seafood as provided in CX/FA 21/52/7 Appendix 5 Annex 2.

Food Additive	**Subcategory for which value was provided**	**Lowest Proposed Residual level (mg/kg)**	**Notes**	**Calculated method performance criteria based on the Lowest Proposed Residual ML**					**Examples of applicable methods that meet the criteria**
				Min Appl. Range (mg/kg)	**LOD (mg/kg)**	**LOQ (mg/kg)**	**Precision (RSD_R (%))**	**Recovery (%)**	
01.6 (*Cheese and analogues)*									
Nitrate	01.6.2.1 *(Ripened cheese, includes rind)*	7	As NO_3	4.5 – 9.5	0.7	1.4	24	80 – 110	**Multi-laboratory validation** - ISO 14673-3 I IDF 189-3: 2004 **Single-laboratory validation -** ISO 14673-2 I IDF 189-2: 2004^
Nitrite	01.6.~~1~~4 *(Processed cheese)* **(see note 1)*	2	As NO_2	1.1 – 2.9	0.2	0.4	29	80 – 110	**Multi-laboratory validation** – not available. **Single-laboratory validation** – not available.
08.0 (*Meat and meat products, including poultry and game)*									
Nitrate	Same residual proposed in multiple food categories including 08.2.1.1 *(Cured (including salted) non-heat treated processed meat, poultry, and game products in whole pieces or cuts)*	7	As NO_3	4.5 – 9.5	0.7	1.4	24	80 – 110	**Multi-laboratory validation** – not available. **Single-laboratory validation** - Afanda, et al.,(2025); Ferreira et al., (2008) for Ham
Nitrite	08.2.1.3 *(Fermented non-heat treated processed meat, poultry, and game products in whole pieces or cuts)*	33	As NO_2	24 – 42	3.3	6.6	19	80 – 110	**Multi-laboratory validation** - EN 12014-3:2005, NMKL 165: 2000 Ed.; AOAC Method 973.31; **Single-laboratory validation** - Afanda, et al., (2025), Ferreira et al., (2008) for Ham; Siu et al., 1998 for Salami, Ham
09.0 (*Fish and fish products, including molluscs, crustaceans, and echinoderms)*									
Nitrite	09.3.3 *(Salmon substitutes, caviar, and other fish roe products)*	4.4	As NO_2	2.7 – 6.1	0.44	0.88	26	80 – 110	**Multi-laboratory validation** – not available. **Single-laboratory validation** – not available.

Notes:

1. The subcategory doesn't match the description in 21/52/7 Appendix 5 Annex 2, as Food category No. 01.6.1 is "Unripened cheese"; while Food Category No 01.6.4 is "Processed cheese".

^. In the absence of LOD or LOQ being specified in the method, the collaborative study report being unavailable at-this-time, and relying on in-house validation data, the validation designated as SLV, although the SLV status may be reviewed with additional data).

Appendix VI

HARMONIZATION OF NAMES FOR PRINCIPLES IN CXS 234-1999

(For further development by the EWG)

1. General Guideline

The name principle mentions only the description of the technique related to determining the test result (Annex A). The techniques used for sample preparation, extraction and separation were not included.

2. Definitions

For the purposes of alignment and harmonization regarding what is considered the principle of an analytical method, the following definition is proposed:

- **Principle** is the technique used for determining the provision, which may include critical information such as, for example, gravimetry - ashing at 550 °C.

To harmonize the descriptions of analytical techniques, the following definitions for main analytical techniques were considered:

- **Biological assay**: It is an analytical method to determine the response, potency or effect of a substance in vivo or in vitro.
- **Calculation**: when the determination is the result of a calculation based on test result(s). In this case, specify the provisions used.
- **Chromatography** is a method used to separate, identify and quantify a component of a mixture by distributing the components between two phases -- stationary phase and mobile phase.
- **Colorimetry**: It is a technique that involves only a colour reaction. The intensity of light (or filtered light) passing through the coloured sample is visually observed or measured and converted to a concentration based on a calibration curve.

  Note: This should not be confused with the tristimulus colorimeter used to measure food colours.
- **Gravimetry**: It is a quantitative analytical method, that is, it determines the amount of a substance by measuring its weight (due to the action of gravity).
- **Potentiometry** is a method of electroanalytical chemistry. It is a quantitative analysis of ions in the solution using measured potentials in an electrochemical cell.
- **Sensory assay**: It is a technique that uses the senses for evaluation of the organoleptic attributes (appearance, odour, texture, taste and others) of a product.
- **Spectroscopy** is a technique which measures electromagnetic radiation for example: UV-Vis (Ultraviolet-Visible) spectrophotometry, infrared, atomic absorption, ICP (Inductively Coupled Plasma), nuclear magnetic resonance (NMR)
- **Mass spectrometry (MS)** is an analytical technique that is used to measure the mass-to-charge ratio of ions.
- **Titrimetry**: It is the determination of a given component from a solution by adding a liquid reagent of known concentration until a given result is achieved.
- **Visual examination**: It is a technique to detect the presence of defects, extraneous or foreign matter in a food through sight, with or without the support of optical equipment (example: magnifying glass, microscope or others).
- **Volumetry**: It is a technique that determines volume without the use of another determining technique, such as titration. In the case of tests where titration is used, it is not called volumetry.

3. Criteria Used

3.1 Assays Whose Results Are Method Dependent

A. Description in the principle of the factor that makes it dependent, if necessary, for example: temperature, conversion factor;

B. Description only of the analytical technique used to obtain the “provision” result, since the other information is described in the methods. Therefore, the following may not be included, unless critical to the “provision” determination, for example: equipment, solvents or reagents used; and

C. For tests that involve the development of microorganisms at a certain temperature, this temperature was included in the “provision” description.

Examples:

- *Moisture at 105 °C – Gravimetry*
- *Protein (Nx6.25) – Titrimetry and calculation*
- *Carbohydrates – Calculation based on the results of moisture, protein, fat, ash and dietary fibre*
- *Artificial dye (qualitative) – Colorimetric*
- *Drained net weight – Gravimetry*
- *Foreign Matter – Visual*
- *Fat – Gravimetry*

3.2 Assays Whose Results Are Independent of the Method

For instrumental tests, the technique used must refer to the main equipment used, for example: for separation, and the detector used for determination.

Examples:

- *Nitrate – UV-Vis (Ultraviolet-Visible) spectrophotometry*
- *Manganese – inductively coupled plasma optical emission spectrophotometry*
- *Potassium – potentiometry with selective electrode*
- *Mercury – atomic absorption spectrophotometry with cold vapor generator*
- *Aflatoxin M1 – high performance liquid chromatography with fluorescence detector*
- *Fatty acids - gas chromatography with flame ionization detector*

4. Additional Information

Considering the acceptance of the criteria described above, it is considered necessary to remove information such as: “ashing”, “ceramic filter filtration”, “complexometry”, “centrifugation”, “weighing”, “distillation”, “enzymatic”, “flotation”, “single sulfation”, “sieving” unless critical to the “provision” determination.

ANNEX A

PRINCIPLES OF METHODS OF ANALYSIS

1. Anodic Stripping Voltammetry (ASV)
2. Atomic Absorption Spectrophotometry (AAS)
   - Cold Vapour (CV AAS)
   - Flame atomic absorption (FAAS)
   - Graphite furnace (GF AAS)
   - Hydride generation (HG AAS)
3. Biological assay
   - Bioassay (in animals, tissue, plants)
   - Microbioassay
4. Immunoassay
   - Enzyme Linked ImmunoSorbent Assay (ELISA)
5. Carbon Isotope Ratio Mass Spectrometry (Carbon IRMS)
6. Centrifugation
7. Colorimetry
8. Conductimetry/Resistivity
9. Confocal Laser Scanning Microscopy (CLSM)
10. Densitometry
11. Detect nuclear DNA Assay
    - DNA Comet Assay
    - Polymerase chain reaction (PCR):
      - PCR conventional (cPCR)
      - Real time qualitative (qPCR)
      - Reverse Transcriptase PCR (RT-PCR)
12. Electrophotometry
    - Electrometric
13. Enzymatic
14. Fluorimetry
15. Gas Chromatography (GC)
    - Electron Capture Detector (ECD)
    - Flame Ionization Detector (FID)
    - Flame Photometric Detector (FPD)
    - Flame Thermionic Detector (FTD)
    - Mass Spectrometry (MS)
    - Nitrogen Phosphorus Detector (NPD)
    - Tandem Mass Spectrometry (MS/MS)
    - Thermal Conductivity Detector (TCD)
    - Quadrupole Time-of-Flight (QTOF)
16. Gravimetry
    - Ashing at (temperature) °C
    - Drying at (temperature) °C
    - Rose-Gottlieb
    - Weibull-Berntrop
    - Schmid-Bondzynski- Ratslaff
    - Vacuum Drying at 70 °C
    - Microwave oven drying
17. Inductively Coupled Plasma (ICP)

- Isotope Dilution Mass Spectrometry (ID MS)
- Mass Spectrometry (MS)
- Optical Emission Spectrometry (OES)
- Quadrupole Inductively couple plasma mass spectrometry (Q-ICPMS)

18. Ion Exchange Chromatography (IC)

- Diode Array Detector (DAD)
- Electrochemical (EC)
- Mass Spectrometry (MS)
- Pulsed Amperometric Detector (PAD)
- Refractive index (RI)
- Conductivity Detector (CD)
- Ultraviolet-Visible (UV/Vis)
- Variable Wavelength Detector (VWD)

19. Liquid Chromatography (LC)

- Diode Array Detector (DAD)
- Fluorescence Detector (FLD)
- High-performance liquid chromatography (HPLC)
- High-Resolution Mass Spectrometry (HRMS)
- Infrared (IR)
- Isotope Dilution Mass Spectrometry (ID MS)
- Mass Spectrometry (MS)
- Matrix-Assisted Laser Desorption Ionization Time of Flight (MALDI-TOF)
- Pulsed amperometry detection (PAD)
- Refractive index (RI)
- Tandem Mass Spectrometry (MS/MS)
- Ultraviolet (UV)

20. Microscopy

- Electronic microscopy
- Optical microscopy

21. Nephelometry

22. Nuclear Magnetic Resonance Spectroscopy (NMR)

23. Photometry

24. Photostimulated Luminescence (PSL)

25. Polarimetry

26. Potentiometry

- Ion selective electrode (EIS)
- Potential of hydrogen pH electrode (pH)

27. Pycnometry

28. Refractometry

29. Spectrometry

- Electron Spin Resonance (ERS)
- Fluorescence (FLD)
- Fourier transform infrared Spectroscopy (FTIR)
- Infrared Spectroscopy (IRS)
- Near Infrared Reflectance Spectroscopy (NIRS)
- Raman (RS)
- Stable isotope mass (IMS)
- Ultraviolet (UV)
- Ultraviolet-Visible (UV-Vis)

30. Thermoluminescence

31. Thermometry

32. Thin Layer Chromatography (TLC)
    - Densitometric detector
    - Fluorescence (FLD)
    - Ultraviolet-Visible (UV-Vis)
33. Titrimetry
    - Acidity
    - Iodimetry & Iodometry
    - Karl Fischer
    - Kjeldahl Digestion
    - Lane & Enyon
    - Wijs
34. Visual examination
35. Volumetry

ANNEX B

ACRONYMS AND ABBREVIATIONS OF PRINCIPLES OF METHODS OF ANALYSIS

AAS	Atomic Absorption Spectrophotometry
AES	Atomic Emission Spectrometry
ASV	Anodic Stripping Voltammetry
Carbon IRMS	Carbon Isotope Ratio Mass Spectrometry
CD	Conductivity Detector
CE	Capillary Electrophoresis
CLSM	Confocal Laser Scanning Microscopy
cPCR	PCR conventional
CVAAS	Cold Vapour Atomic Absorption Spectrophotometry
DAD	Diode Array Detector
EC	Electrochemical Detector
ECD	Electron Capture Detector
EIS	Ion selective electrode
ELISA	Enzyme Linked ImmunoSorbent Assay
ESR	Electron Spin Resonance
FAAS	Flame Atomic Absorption Spectrophotometry
FIA- AAS	Flow injection Analysis Atomic Absorption Spectrophotometry
FID	Flame Ionization Detector
FLD	Fluorescence Detector
FPD	Flame Photometric Detector
FTD	Flame Thermionic Detector
FTIR	Fourier transform infrared spectroscopy
GC	Gas Chromatography
GFAAS	Graphite furnace Atomic Absorption Spectrophotometry
HGAAS	Hydride generation Atomic Absorption Spectrophotometry
HPLC	High Performance Liquid Chromatograph
HPTLC	High Performance Thin Layer Chromatography
HRMS	High-Resolution Mass Spectrometry
IC	Ion Chromatography
ICP	Inductively Coupled Plasma
ID	Isotope Dilution
IMS	Stable isotope mass
IR	Infrared
IRS	Infrared Spectroscopy
LC	Liquid Chromatograph
MALDI	Matrix-Assisted Laser Desorption Ionization
MS	Mass Spectrometry
MS/MS	Tandem Mass Spectrometry

NIRS	Near Infrared Reflectance Spectroscopy
NMR	Nuclear Magnetic Resonance Spectroscopy
NPD	Nitrogen Phosphorus Detector
OES	Optical Emission Spectrometry
PAD	Pulsed Amperometry Detection
PCR	Polymerase chain reaction
pH	pH electrode
PSL	Photostimulated Luminescence
qPCR	Real Time Qualitative
Q-ICPMS	Quadrupole Inductively couple plasma mass spectrometry
QTOF	Quadrupole Time-of-Flight
RI	Refractive Index
RS	Raman Spectroscopy
RT-PCR	Reverse Transcriptase PCR
TLC	Thin-layer chromatography
TOF	Time of Flight
UHPLC	Ultra-High Performance Liquid Chromatograph
UV	Ultraviolet
UV-Vis	Ultraviolet-Visible
VWD	Variable Wavelength Detector

ANNEX C

LIST OF ACRONYMS FOR STANDARD METHOD REFERENCES

AACC	Cereals & Grains Association	(www.cerealsgrains.org/)
AIIBP	International Association of the Bouillon and Soup Industry	(www.culinaria-europe.eu/)
Anal. Chim. Acta.	Analytica Chimica Acta	(https://www.sciencedirect.com/journal/analytica-chimica-acta)
AOAC	AOAC International	(www.aoac.org/)
AOCS	American Oil Chemists' Society	(www.aocs.org/)
BS	British Standard	(www.bsigroup.com)
COI	Collection of methods by the International live	(www.internationaloliveoil.org/)
EN	European Standards	(www.en-standard.eu/)
EPA	Environmental Protection Agency	(www.epa.gov/)
EUsalt	European Salt Producers Association	(https://eusalt.com/)
FDA	Food and Drug Administration [Laboratory methods]	(www.fda.gov/)
ICC	International Association for Cereal Science and Technology	(https://icc.or.at/)
ICUMSA	International Commission for Uniform Methods of Sugar Analysis	(www.icumsa.org/)
IDF	International Dairy Federation	(https://fil-idf.org/)
IFU	International Fruit and Vegetable Juice Association [IFU Methods Analysis IFUMA]	(https://ifu-fruitjuice.com/)
IHC	International Honey Commission	(www.ihc-platform.net/)
ICA	International Office of Cocoa, Chocolate, and Sugar Confectionery	(www.icco.org/)
IS	Indian Standard	(www.bis.gov.in/)
ISI	International Starch Institute	(www.starch.dk/)
ISO	International Organization for Standardization	(www.iso.org/)
IUPAC	International Union of Pure and Applied Chemistry	(www.iupac.org/); (www.old.iupac.org/)
NMKL	Nordic-Baltic Committee on Food Analysis	(www.nmkl.org/)
OIV	International Organisation of Vine and Wine	(www.oiv.int/)
Ph. Eur	European Pharmacopoeia	(https://www.edqm.eu/en/the-european-pharmacopoeia)
USP	US Pharmacopeia	(www.usp.org/)
WEFTA	West European Fish Technologists Association	(www.wefta.org)