codex alimentarius commission

FOOD AND AGRICULTURE ORGANIZATION OF THE UNITED NATIONS

WORLD HEALTH ORGANIZATION

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ALINORM 03/23

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX ALIMENTARIUS COMMISSION

Twenty-sixth Session
Rome, Italy, 30 June - 5 July 2003

REPORT OF THE TWENTY-FOURTH SESSION OF THE CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

Budapest, Hungary
18 - 22 November 2002

Note: This document incorporates Codex Circular Letter CL 2002/53-MAS

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CX 4/50.2 **CL 2002/53-MAS**
November 2002

TO: - Codex Contact Points
- Interested International Organizations

FROM: - Secretary, Codex Alimentarius Commission, Joint FAO/WHO Food Standards Programme, FAO, 00100 Rome, Italy

SUBJECT: **Distribution of the Report of the 24th Session of the Codex Committee on Methods of Analysis and Sampling (ALINORM 03/23)**

A. MATTERS FOR ADOPTION BY THE 26th SESSION OF THE CODEX ALIMENTARIUS COMMISSION

PROPOSED AMENDMENTS TO THE PROCEDURAL MANUAL

1. Amendment to the *General Criteria for the Selection of Methods of Analysis Using the Criteria Approach* and new section on *Working Instructions for the Implementation of the Criteria Approach in Codex* (para. 42, Appendix II)

GUIDELINES FOR ADOPTION BY REFERENCE FOR CODEX PURPOSES

2. Harmonized IUPAC *Guidelines for Single-Laboratory Validation of Methods of Analysis* (para. 95, Appendix III)

METHODS OF ANALYSIS AND SAMPLING

3. General Methods of Analysis for Additives, Contaminants and Irradiated Foods (paras. 61-66, Appendix VI - Sections G. and H.)

4. Methods of Analysis in Commodity Standards at different steps (paras. 57-60 and 67-70, Appendix VI - Sections A. to F.)

Governments wishing to propose amendments or comments on the above documents should do so in writing in conformity with the Guide to the Consideration of Standards at Step 8 (see Procedural Manual of the Codex Alimentarius Commission) to the Secretary, Joint FAO/WHO Food Standards Programme, FAO, Viale delle Terme di Caracalla, 00100 Rome, Italy **before 15 March 2003.**

PROPOSED DRAFT GUIDELINES AT STEP 5

5. Proposed Draft General Guidelines on Sampling (para. 19, Appendix IV)

6. Proposed Draft Guidelines on Measurement Uncertainty (para. 52, Appendix V)

Governments wishing to submit comments on the implications which the Proposed Draft Amendment may have for their economic interests should do so in writing in conformity with the Procedure for the Elaboration of World-wide Standards at Step 5 to the Secretary, Codex Alimentarius Commission, Joint FAO/WHO Food Standards Programme, FAO, via delle Terme di Caracalla, 00100 Rome, Italy **before 15 March 2003**.

B. REQUEST FOR COMMENTS AND INFORMATION

7. Proposed Draft Guidelines for Evaluating Acceptable Methods of Analysis (para. 26, Appendix VII)

Governments and international organizations wishing to submit comments at Step 3 should do so in writing to the Secretary, Joint FAO/WHO Food Standards Programme, FAO, Viale delle Terme di Caracalla, 00100 Rome, Italy, with a copy to Dr. Mária Váradi, Central Food Research Institute (KÉKI), H-1022 Budapest, Herman Ottó út 15 (Fax No., +361.212.9853 & 361.355.8928; e-mail, m.varadi@mail.cfri.hu **before 30 May 2003.**

SUMMARY AND CONCLUSIONS

The summary and conclusions of the 24th Session of the Codex Committee on Methods of Analysis and Sampling are as follows:

Matters for consideration by the Commission:

The Committee:

- agreed to propose the inclusion of a new section on *Working Instructions for the Implementation of the Criteria Approach in Codex* in the Procedural Manual and a consequential amendment to the *General Criteria for the Selection of Methods of Analysis Using the Criteria Approach* (para. 42, Appendix II);
- proposed that the Commission adopt by reference for Codex purposes the *Harmonized IUPAC Guidelines for Single-Laboratory Validation of Methods of Analysis* (para. 95, Appendix III);
- endorsed several methods of analysis in commodity standards at different steps of the Procedure; and proposed several methods for additives, contaminants and the detection of irradiated foods for adoption as general Codex methods (paras. 57-70, Appendix VI);
- advanced to Step 5 the Proposed Draft General Guidelines on Sampling (para. 19, Appendix IV);
- advanced to Step 5 the Proposed Draft General Guidelines on Measurement Uncertainty (para. 52, Appendix V);
- agreed to initiate new work on Proposed Draft Guidelines for Settling Disputes on Analytical (Test) Results (para. 32) and on the review of the current *Analytical Terminology for Codex Use* in the Procedural Manual (para. 95).

Other Matters of Interest to the Commission

The Committee:

- agreed to return the Proposed Draft Guidelines for Evaluating Acceptable Methods of Analysis to Step 3 (para. 26, Appendix VII);
- agreed to consider criteria for methods of analysis for foods derived from biotechnology at its next session (para. 81).

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INTRODUCTION

1. The Codex Committee on Methods of Analysis and Sampling held its Twenty-fourth Session in Budapest, Hungary, from 18 to 22 November 2002, by courtesy of the Government of Hungary. The Session was chaired by Professor Peter Biacs, Deputy State Secretary of the Ministry of Agriculture and Regional Development and by the Vice-Chairperson, Prof. Pal Molnar, Head of Food Quality Department of the Central Food Research Institute (KEKI). The Session was attended by 135 delegates and observers representing 46 Member Countries and 12 international organizations. A complete list of participants is given in Appendix I of this report.

OPENING OF THE SESSION

2. The Session was welcomed by Dr Tibor Szanyi, Parliamentary State Secretary of Ministry of Agriculture and Regional Development. Dr Szanyi emphasized the role of the Codex Alimentarius standards in assuring food safety and their importance for harmonization and international food trade. He indicated that Hungary had been very pleased to host this Committee for thirty years and informed the participants about the Government's initiatives in ensuring food safety in Hungary especially the decision of the Hungarian Government to establish a Food Safety Office. Dr Szanyi stressed the importance of the work of the Committee on Methods of Analysis and Sampling in ensuring compliance with provisions in Codex standards and wished the delegates all success in their work.

ADOPTION OF THE AGENDA (Agenda Item 1)

3. The Committee decided to postpone the consideration of Agenda Item 3 to a later time in order to allow the participants additional time to study the amendments made by the *Ad Hoc* Working Group held prior to the session, as suggested by the Delegation of France. It also agreed to consider Agenda Items 4 b) and 7 b) before items 4 a) and 7 a) respectively as they contained more general issues on which the Committee had to agree before entering into specific discussions. With these amendments the Committee adopted the Provisional Agenda as presented in CX/MAS 02/01.

MATTERS REFERRED BY THE CODEX ALIMENTARIUS COMMISSION AND OTHER CODEX COMMITTEES (Agenda Item 2)[1]

4. The Committee noted that a number of matters referred by the 24th Session of the Codex Alimentarius Commission (CAC), Executive Committee and other Codex Committees were for information purposes or would be discussed in more detail under the relevant Agenda items. In addition the Committee noted matters referred as follows:

Methods for dioxins and PCBs

5. The Delegation of Germany informed the Committee that the document on the determination of dioxins and PCBs had not been prepared as there were no proposals received from Member Governments before this Session. The Committee agreed that a Circular Letter would request Member Governments and interested international organizations to submit their proposals for the determination of dioxins and PCBs to Germany who would prepare a paper for consideration at the next session of the Committee.

Chloramphenicol in Shrimps

6. The Committee noted the referral from the 13th Session of the FAO/WHO Regional Coordinating Committee for Asia (ALINORM 03/15, paras 151-155) that there was a need to give attention to the resolution of the problem of abrupt changes in analytical techniques, and changes in detection limits at the level of determination and was informed that the comments of India presented in CRD 6 could be taken into consideration from a general point of view on the relevant Agenda Items of this Committee.

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1 CX/MAS 02/02; CX/MAS 02/2-Add.1.

PROPOSED DRAFT GENERAL GUIDELINES ON SAMPLING (Agenda Item 3)[2]

7. The Committee recalled that at its 23rd Session it had been agreed that the Delegation of France would continue its work with the assistance of a drafting group working by electronic mail in order to complete the revision of the text for circulation at Step 3 and that a Working Group would meet prior to the 24 th Session in order to incorporate comments received and facilitate discussion in the Plenary.

8. The Delegation of France presented the progress report on the Proposed Draft Guidelines between the 23rd and current Session and introduced CRD 16 prepared by the *Ad Hoc* Working Group. The Delegation pointed out that following the decisions of the last session of the Committee, the Drafting Group had prepared a self-explanatory, simplified document that followed the scientific statistical approach and incorporated written comments which had not been taken into account by the last session of the Committee. The Delegation informed the participants that the Working Group that had met prior to this session, had revised the document in the light of comments received and prepared the above CRD for consideration by the Plenary. The Delegation indicated that all additional changes were highlighted in CRD 16 for easy reference and that the following major new amendments were made:

- the Preamble was divided into two parts, in place of the foreword;
- Scope and Table 1 were amended for clarification purposes;
- New sections 3.3 and 4.4 on special sampling for average controls were inserted;
- Three Tables from NMKL Procedure 12 describing the number of items to be sampled at different inspection levels were introduced to make the Proposed Draft Guidelines easier to use.

9. The Committee considered the Proposed Draft Guidelines presented in CRD 16 section by section and made the following amendments in addition to several editorial changes throughout the text.

10. The Committee inserted "for example" in footnote 2 in order to clarify the use of a pragmatic approach in the Committee on Pesticide Residues (CCPR) and the Committee on Residues of Veterinary Drugs in Foods (CCRVDF).

11. In Section 1.4 Scope of the Guidelines the Committee substituted measurement "uncertainty" by measurement "error" and added wording "sampling error" with the understanding that these terms would be changed accordingly and further clarified in Section 2.4 on Estimation Errors.

12. The Committee clarified in the Scope that these Guidelines are mainly applicable for the control at reception, and may not be applicable for the control of end products and for process control during production.

13. In Section 1.5 the Committee accepted the proposals of ISO and corrected the references to several of their standards.

14. In Section 2.4, it was agreed to include both a first "specific case" with measurement error of the same order of magnitude than sampling error, and a "second specific case" where measurement error is larger than sampling error and there is no need for statistical sampling plans. It was also agreed that the Guidelines did not consider how to take measurement error into account.

15. The Committee accepted the proposal of the Delegation of Indonesia and amended the next to final paragraph in Section 2.6 to indicate that the choice of plans corresponding to low AQL values depended on the product.

16. The Committee deleted the end of the last sentence on the complexity of sampling plans in Section 2.6 and made a reference to the relevant ISO standards.

17. The Committee inserted the wording "Procedure" and deleted the text in brackets in the first column under the Lot size in Tables 10, 14 and 17 for clarification purposes.

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[2] CX/MAS 02/3; CX/MAS 02/3-Add.1 (comments of Canada, Czech Republic, Hungary, South Africa and United Sates); CX/MAS 02/3-Add.2 (comments of New Zealand); CX/MAS 02/3-Add.3 (comments of France); CRD 3 (version prepared for consideration by the *Ad Hoc* Working Group); CRD 7 (comments Brazil, India); CRD 13 (comments from Philippines); CRD 14 (comments from Australia), CRD 16 (version prepared by the *Ad Hoc* Working Group for consideration by the Plenary)

18. The Committee expressed its warm appreciation to the Delegation of France and the members of the Working Group for their excellent work in this Session and in recent years that had allowed the Committee to make considerable progress on complex and long-standing issues. The Committee recognized that the development of the Guidelines would provide important guidance to governments and other uses of sampling plans. The Committee agreed to convene the Working Group again prior to the next Session to review the comments and facilitate discussions on the finalisation of the document.

Status of the Proposed Draft General Guidelines on Sampling

19. The Committee agreed to advance the Proposed Draft General Guidelines on Sampling, as amended during this Session, to Step 5 of the Procedure for adoption by the 26th Session of the Commission (see Appendix IV).

CRITERIA FOR EVALUATING ACCEPTABLE METHODS OF ANALYSIS FOR CODEX PURPOSES (Agenda Item 4)

PROPOSED DRAFT GUIDELINES FOR EVALUATING ACCEPTABLE METHODS OF ANALYSIS (Agenda Item 4a)[3]

20. The last session of the Committee had agreed to develop guidelines for evaluating methods of analysis intended for governments, in addition to the criteria for inclusion in the Procedural Manual. The Executive Committee had subsequently approved this proposal for new work but the Proposed Draft Guidelines had not been circulated for comments due to lack of time.

21. The Delegation of the United Kingdom highlighted the possible approaches that could be applied to the complex question of evaluating methods of analysis: to identify specific performance parameters and assign numeric values to these according to the traditional approach (Appendix II of the document); or to identify a "fitness for purpose" approach, taking all values into account by defining a single parameter or fitness function (Appendix I). Fitness functions could describe the actual performance of a specific method ("characteristic function") and the uncertainty that is fit for purpose for a specific field of application. The Delegation noted that the development of this relatively new concept would require substantial work but it would be especially relevant in the light of the performance-based approach to the selection of methods.

22. The Delegation of the Philippines supported this approach as it would give the possibility to developing countries to select methods on the basis of their fitness for purpose and it allowed to select methods with a graphic comparison of the characteristic function with the fitness function.

23. Several delegations expressed the view that the "fitness for purpose" approach was interesting and should be considered in more detail in the future. However, this might be a long-term process, and at this stage the Committee should provide guidance to governments according to the traditional approach.

24. Some delegations proposed to include examples in the Guidelines in order to facilitate their practical application. It was noted that examples might not appear in the final text of the Guidelines, that would include general recommendations. However, they could be used in the elaboration process and would be available in working documents as a reference for member countries.

25. The Committee agreed that, in order to facilitate progress, the Proposed Draft Guidelines reflecting the "traditional approach" (Appendix II of the document) should be circulated for comments at Step 3, while the section on "fitness for purpose" would be redrafted for further consideration at the next session. The Committee expressed its appreciation to the Delegation of the United Kingdom for its comprehensive work on these complex issues.

Status of the Proposed Draft Guidelines for Evaluating Acceptable Methods of Analysis

26. The Committee agreed to circulate the Proposed Draft Guidelines for comments at Step 3 (see Appendix VII). The Committee also agreed that the Delegation of the United Kingdom, with the assistance of a drafting group[4] would revise the text in the light of the comments received and would also develop the document on the "fitness for purpose" approach, for further consideration at the next session.

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[3] CX/MAS 02/4, CX/MAS 02/4-Add.2 (document on dispute situations prepared by France), CRD 8 (comments of India), CRD 13 (comments of the Philippines), CRD 17 (Proposal for new work on dispute situations)

[4] Austria, Finland, France, Japan, Netherlands, Switzerland, United States

DISPUTE SITUATIONS

27. The Committee recalled that its last session had discussed dispute situations in the framework of the criteria approach and had agreed that the Delegation of France, in cooperation with other countries, would prepare a document addressing this question.

28. The Delegation of France presented a document based on some sections of ISO 4259:2000, and proposing procedures for settling interlaboratory disputes, in the absence of specific rules set out in the specification or mentioned in the test method. The document provided a step by step approach to identify the causes of disagreement between laboratories on analytical results and facilitate their settlement. On this basis, the Delegation proposed to initiate new work on guidelines for dispute settlement.

29. Some delegations expressed the view that although the document followed a scientific approach, it was too complex for the purposes of Codex and a simpler and more practical approach should be followed, as proposed in the written comments of Thailand. Reference was also made to the earlier recommendations of the Committee on Food Import and Export Inspection and Certification Systems to the effect that recommendations in this area should not be too prescriptive[5].

30. After some discussion, it was agreed that the Delegation of France, in cooperation with the delegations of Australia, Canada, Finland, New Zealand, Netherlands, Sweden, United Kingdom, United States would work during the session to propose a revised outline. The result of these discussions was presented to the Committee in CRD 17 "Proposal for New Work on Dispute Situations".

31. The Committee welcomed this proposal and recognized that disputes might arise from differences due to sampling; differences in the analytical procedures; and differences in the interpretation of test results. Some delegations supported considering all types of disputes, including the differences relating to sampling plans. However the Committee agreed to concentrate on the settlement of differences in analytical procedures at this stage, and to develop guidelines that would deal with two situations 1) the same validated method is used by both laboratories; 2) two different validated methods are used by each laboratory. The guidelines would specify how this apparent disagreement could be resolved step by step.

32. The Committee agreed to initiate new work on Proposed Draft Guidelines for Settling Disputes over Analytical (Test) Results, to be developed by the Delegation of France in cooperation with a Drafting Group[6] for consideration by the next session, subject to the approval of the Commission.

PROPOSED AMENDMENTS TO THE PRINCIPLES FOR THE ESTABLISHMENT OF CODEX METHODS OF ANALYSIS (Agenda Item 4b)[7]

33. The Committee recalled that its last session had approved in principle the criteria approach and proposed amendments to the *Principles for the Establishment of Codex Methods of Analysis* and to the *Relations between Commodity Committees and General Committees* that were subsequently adopted by the Commission and included in the Procedural Manual. In addition the CCMAS had proposed Working Instructions for the Implementation of the Criteria Approach. The Commission had agreed that the simplified text prepared by the Delegation of Sweden, in cooperation with Japan and the United Kingdom, should be referred back to the Committee on Methods of Analysis and Sampling for further consideration.

34. The Delegation of the United Kingdom recalled the progress achieved with the adoption of the criteria approach and stressed the importance of providing working instructions to Codex Committees to facilitate its application. This view was supported by several delegations.

35. The Delegation of Japan supported the adoption of the text, with the exception of the Retroactive Action, and expressed some reservations about the inclusion of definitions as the terminology proposed was still under discussion in member countries and at the international level.

36. Other delegations pointed out that definitions were necessary in the framework of the criteria approach, with the understanding that they were subject to further review, and asked for clarification in this respect.

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5 ALINORM 01/30, para. 101

6 Australia, Canada, Finland, New Zealand, Netherlands, Philippines, Sweden, Switzerland, United Kingdom, United States

7 CX/MAS 02/5, CX/MAS 02/5-Add. 1 (comments of France, EC), CX/MAS 02/5-Add.2 (comments of United States), CRD 8 (comments of India)

The Secretariat indicated that definitions in the Procedural Manual could be revised regularly and that a note could be included to specify that they were adopted on an interim basis and subject to revision, as in the case of the definitions for risk analysis. The Committee agreed to include a note to this effect in the Analytical Terminology and to revise it regularly as required.

37. The Committee agreed to proceed as follows as regards Retroactive Action. The methods already adopted by Codex should be left as at present and the criteria approach should be applied only to methods that are still to be elaborated in Codex standards or endorsed by CCMAS, except in cases where a multiplicity of methods are considered for endorsement as Type III methods. However, it was not necessary to include a section to this effect in the *Working Instructions*.

38. The Delegation of Denmark, speaking on behalf of the Member States of the European Union, proposed to apply the criteria approach to Type II methods, in addition to Type III. The Delegation of the United States, while supporting the criteria approach in principle for both Types, expressed the view that Type II methods should not be eliminated before dispute situations had been addressed, and also proposed to include examples to clarify its application.

39. Several delegations pointed out that from the scientific point of view, there was no difference in the consideration of Type II and Type III methods and that the same criteria should apply to both. It was also noted that Type II methods were selected from Type III methods.

40. The Committee recalled that the text proposed was not prescriptive and left the possibility to Codex Committees or to the CCMAS to select either a specific method or criteria. This would apply to both Types and it would be the responsibility of the Committee to endorse the method or the criteria according to the provision and analytes concerned on a case by case basis. After an exchange of views, the Committee agreed that the criteria approach would apply to Type II and Type III methods. The Committee therefore agreed to amend the *General Criteria for the Selection of Methods of Analysis using the Criteria Approach* (adopted in 2001) to reflect that they applied also to Type II methods.

41. As regards the general requirements for methods, the Committee discussed whether Type III methods should be collaboratively tested. Some delegations indicated that this was not always possible, especially for trace elements. It was noted that this would be discussed more specifically under Agenda Item 8 but that in principle collaborative studies should be required for Type III methods.

Status of the Proposed Amendments to the Principles for the Establishment of Codex Methods of Analysis (Working Instructions for the Implementation of the Criteria Approach)

42. The Committee agreed to forward the proposed *Working Instructions for the Implementation of the Criteria Approach* and the consequential amendment to the *General Criteria for the Selection of Methods of Analysis using the Criteria Approach* to the Committee on General Principles for endorsement and to the Commission for adoption and inclusion in the Procedural Manual (see Appendix II).

PROPOSED DRAFT GUIDELINES ON MEASUREMENT UNCERTAINTY (Agenda item 5)[8]

43. The Committee recalled that its last Session had agreed to circulate the *Proposed Draft Guidelines on Measurement Uncertainty* for comments at Step 3 (Appendix V, ALINORM 01/23 by CL 2001/5-MAS), subject to approval as new work. The elaboration of the *Proposed Draft Guidelines* had been approved as new work by the 49th (Extraordinary) Session of the Executive Committee (ALINORM 03/3, para. 21, Appendix III).

General aspects

44. The Delegation of Malaysia, supported by other delegations, expressed the view that the term "measurement uncertainty" was widely used whereas "measurement reliability" had not been defined yet. The Committee agreed to delete the term "measurement reliability" and all square brackets related to this terminology throughout the text.

Introduction

45. The Committee recognized that the recommendations concerning uncertainty included in the first sentence were a requirement under ISO/IEC 17025:1999 and amended the text accordingly.

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8 ALINORM 01/23, Appendix V, CL 2001/5-MAS , CX/MAS 02/6, CX/MAS 02/6-Add.1 (comment of New Zealand, Spain and Thailand), CX/MAS 02/6-Add.2 (comment of United States), CRD 9 (comment of Brazil, European Commission) and CRD 13 (comment of Philippines)

46. The Delegation of New Zealand, supported by the Delegation of Australia, proposed to specify that "any single analytical test result relates only to the single sample, and a statistically valid sampling plan must be utilised" in order to avoid the possible misuse of the Guidelines. However, some delegations stressed that the Guidelines were intended to cover only measurement uncertainty, not sampling uncertainty, and the current text was retained. The Committee also noted that the issues related to sampling and measurement uncertainty would be discussed from a more comprehensive perspective under Agenda Item 9.

47. The Committee agreed with the proposal of the Delegation of New Zealand to add a footnote addressing the need to find a satisfactory surrogate for reproducibility when inter-laboratory studies were not possible.

48. The Committee agreed to clarify the requirement that laboratories should be "in control", as proposed in the written comments of Brazil. A footnote referring to the *Guidelines for the Assessment of the Competence of Testing Laboratories Involved in the Import and Export Control of Food*" (GL 27-1997) was included for this purpose, as proposed by the Delegation of the United Kingdom. The Committee noted that the reference to ISO/IEC Guide 25 had been superseded by ISO/IEC 17025:1999 and agreed that the reference in the Guidelines (GL 27-1997) should be updated accordingly.

Recommendations

49. In Recommendation 2, the Delegation of New Zealand proposed that where an estimate of the uncertainty was made available, the basis by which it was derived should be included. However, this was not accepted as some delegations pointed out that such information should be made available only when required by the customer, according to current practice.

50. The Delegation of New Zealand, supported by Australia, proposed to add a new recommendation in order to require the annotation of the results as corrected or uncorrected and the estimation of the recovery factor applied. However, the Committee noted that recovery factors were already covered by Codex recommendations[9] and agreed that the Guidelines should provide clear and specific guidance on measurement uncertainty.

51. The Delegation of Ireland, while supporting the current objectives of the Guidelines, pointed out that measurement uncertainty, recovery factors and other related issues that had been discussed individually should be considered in an overarching framework as they all affected the use of analytical results.

Status of the Proposed Draft Guidelines on Measurement Uncertainty

52. The Committee agreed to forward the Proposed Draft Guidelines to the Commission for adoption at Step 5 of the Procedure (see Appendix V).

ENDORSEMENT OF METHODS OF ANALYSIS PROVISIONS IN CODEX STANDARDS (Agenda Item 6)[10]

53. The report of the *Ad hoc* Working group on Endorsement of Methods of Analysis (CRD 1) held prior to the session was presented by its Chair, Dr. Pal Molnar (Hungary).

General issues

54. The Committee confirmed that the methods proposed by Codex commodity and general committees for endorsement should correspond to provisions in Codex standards or standards in the elaboration procedure. The Committee agreed that in application of the procedure, the methods that did not correspond to a specific provision could not be considered for endorsement. In particular, the Committee did not consider the methods forwarded by the *Ad hoc* Intergovernmental Task Force on Fruit and Vegetable Juices and asked the Task Force to identify the methods corresponding only to specific provisions in the Proposed Draft Standards under elaboration.

55. The Committee recalled that when equivalent methods exist, they should all be listed, and the organisations concerned were invited to provide the relevant references for inclusion in the list of methods.

56. The Committee recognized that all information on the validation of methods should be available and that methods should be fully traceable, as it was important to check that this information existed and was available to users, when deciding on the endorsement of methods.

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9 IUPAC Guidelines for the Use of Recovery Information in Analytical Measurement (CAC/GL 37-2001)

10 CX/MAS 02/7, CX/MAS 02/7-Add.1, CX/MAS 02/7-Add.2, CRD 1 (report of the Working Group), CRD 8 (comments of India), CRD 10 (comments of Argentina)

Fats and Oils

57. As regards methods for Fat Spreads and Blended Spreads, AOAC 986.15 was retained as the only method for the determination of arsenic as the other methods were not any longer available, and it was noted that endorsement would be subject to the finalization of a maximum limit for arsenic. The Committee on Fats and Oils was also asked for clarification on the calculation to determine "milk fat" as the method proposed applies to butyric acid.

Chocolate and Chocolate Products

58. The Committee endorsed the method proposed for the determination of vegetable fat in chocolate and chocolate products, with an update of reference to the AOCS method for the detection of breakdown sterol products in refined vegetable fats.

Milk and Milk Products

59. The Committee noted that there was no reply from the Committee on Milk and Milk Products to some earlier questions and did not endorse the methods concerned, pending further clarification from the Committee. Method AOAC 947.05 was temporarily endorsed, pending clarification on the type of the method required. The other methods were endorsed and additional references to equivalent methods were added where necessary. The Committee endorsed the equivalent methods proposed as Type I for milk fat in dried matter for cottage cheese only, noting that other methods had been endorsed as Type I for individual cheeses.

Fish and Fishery Products

60. The Committee noted that the method for water activity applied to canned vegetables and asked for clarification on its use for boiled dried salted anchovies. The Delegation of Finland indicated that there was an NMKL method for the determination of water activity (NMKL 168(2001)). The method for the determination of acid insoluble ash was referred back to the Committee on Fish and Fishery Products for further information on the validation of the method.

Irradiated Foods

61. The Committee recalled the discussions of the Commission concerning the availability of methods and the possibility to use them in developing countries. The Observer from the EC informed the Committee that three of the new methods were easy to use and did not require costly equipment, and also recalled that all methods had been fully validated.

62. The Delegation of Australia, referring to the written comments of Argentina, expressed the view that the results were not representative of the actual percentage of false positives and negatives for irradiated and non-irradiated foods (method EN 13784:2001). The Observer from the EC explained the results of the interlaboratory studies, in particular regarding the occurrence of false positives and false negatives.

63. The Committee endorsed the four new methods proposed and replaced the general method EN 1788:1996 with its updated version EN 1788:2001. The reference to the NMKL method 137 (2002) that had been validated for raw minced meat was also added to method EN 13783:2001.

Additives and Contaminants

64. The Committee endorsed the methods for additives and contaminants corresponding to specific provisions under consideration or included in adopted standards. Method EN 12955:1999-07 was endorsed as it applies to the sum of aflatoxins in peanuts, for which a maximum level has been established[11]. The Committee recalled that several methods had been endorsed earlier for aflatoxins and they were included in the Table for reference. After some discussion on the need for amendments to the type of the current methods, it was agreed to retain AOAC 991.31 for total aflatoxins in raw peanuts as Type II and to endorse EN 12955:1999-07 as Type III.

65. The Committee agreed to delete the method for aflatoxin in maize that had been endorsed earlier as there was no maximum level for aflatoxin in maize.

66. The Committee, recalling that methods for cyclamate and saccharin had been endorsed earlier[12], considered whether changes were required to the endorsement status. It was agreed to retain NMKL

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11 CODEX STAN 209-1999: 15 µg/kg for total aflatoxins in peanuts intended for further processing

12 ALINORM 97/23A, Appendix V

122(1997) for saccharin in beverages and sweets as Type II and to endorse EN 12856: 1999-04 for saccharin in all foods as Type III. As regards cyclamates, the Committee endorsed EN 12857:1999-04 as Type II and retained the current NMKL method 123 (1998) as Type III. The methods proposed for nitrates/nitrites in meat products were temporarily endorsed pending final publication of the validation results.

Processed Fruits and Vegetables

67. The Committee asked for clarification from the Committee on Processed Fruits and Vegetables on the provision and/or commodity concerned by the determination of pH and sulphites. It was noted that a general method for sulphites had been endorsed and that it applied to processed fruits and vegetables. The Committee also recommended that the Commodity Committee consider ISO 1842:1991 as it was specific for pH in processed fruits and vegetables, if the determination of pH was required in a standard under consideration.

68. The Committee asked for clarification on the amendment proposed to AOAC 968.30 for the determination of drained weight, and on how sections 2.1 and 2.2 should be amended.

69. The Committee did not endorse the methods for moisture, non-fat solids, total fat and total solids for aqueous coconut products as the methods applied to milk.

70. The Committee deleted the methods for acidity, salt and drained weight for pickles as no relevant provisions existed in the Draft Standard. It recalled that the method proposed as Type IV for lead was temporarily endorsed since 1998 and asked the Commodity Committee whether this method was necessary since a general Codex method already existed as Type II. As regards the determination of benzoic acid and sorbates, it was recommended that the Committee consider more modern methods (liquid chromatography) such as NMKL 124 (1997).

CONSIDERATION OF METHODS FOR THE DETECTION AND IDENTIFICATION OF FOODS DERIVED FROM BIOTECHNOLOGY: GENERAL APPROACH AND CRITERIA FOR THE METHODS (Agenda Item 7 b)[13]

71. The Delegation of Germany introduced the document and indicated that following the request from the Committee on Food Labelling and the Task Force on Foods Derived from Biotechnology to consider the methods of analysis for foods derived from biotechnology Germany and the United Kingdom had prepared the paper describing the current status of methodology and the problems to be addressed. The Delegation informed the Committee that the presence of a genetically modified organism or its derivatives could be accomplished by the detection of either DNA sequences present as a result of recombination or the protein coded by the inserted gene.

72. The Delegation pointed out that protein based methods were cheap, offered high selectivity and sensitivity, however since proteins were denatured during processing these techniques were mostly suitable for raw material analysis and not applicable to highly processed foods. It was also noted that these methods cannot be used when no protein is expressed in the food. Methods for detection of DNA markers based on the Polymerase Chain Reaction (PCR) had been used in a variety of food analyses and widely used for detection of GM derivatives in food for many years, and the modifications of the PCR method were also widely used. A typical method involved several steps such as extraction and purification, amplification by PCR and detection / quantification. In this regard the Delegation drew the attention of the Committee to specific questions arising in the area of proficiency testing, use of performance criteria and the necessity of quantification of the threshold since the results of investigations showed the difficulties in measuring low levels of GM material in processed foods. The Delegation pointed out that the methods described in the document could be used if all information about the sequence and standard reference materials were available.

73. In view of the absence of precise provisions for GMOs and of difficulties with the practical application of methodology in this area the Delegation proposed to develop recommendations with respect to quality control measures in laboratories offering GM analyses and specific criteria for methods of analysis.

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[13] CX/MAS 02/9; CRD 5 and CRD 15 (comments of France); Information paper submitted by Japan containing two analytical methods on GMOs „Novel Reference Molecules for Quantitation of Genetically Modified Maize and Soybean", Journal of AOAC International, Vol. 85, No 5, 2002 and „Validation of Real-Time PCR Analyses for Line-Specific Quantitation of Genetically Modified Maize and Soybean Using New Reference Molecules", Journal AOAC International, Vol. 85, No 5, 2002.

74. The Delegation of France drew the attention of the Committee to the basic problems still existing in relation to the double source of uncertainty, one arising from measurement, the other from the calibration curve used. The Delegation also drew the attention of the Committee to CRD 5 and 15 proposing criteria that could be used for the control of GMOs in processed foods.

75. The Delegation of the United Kingdom was of the view that the paper presented was a very good start describing currently used methodologies for the detection of GM and pointed out that in view of forthcoming methodological developments such as the use of mass spectrometry, there was a need to work continuously in this area. The Delegation pointed out that GM analysis was very difficult in view of the very small amount of measured analyte especially in certain foods such as biscuits, and that fundamental methodological problems in relation to distribution of results, collaborative trials, availability of reference materials should be solved before proceeding further. The Delegation stressed that the criteria approach could be most suitable in this area of analysis.

76. The Delegation of Ireland drew the attention of the Committee to the existence of the network of GMO investigating laboratories and stressed the necessity of better partnership between the regulatory agencies and industry in this specific area.

77. Several delegations stressed the difficulties related to the availability of reference materials and the Committee noted that the access to GM reference materials was of prime importance for GM analysing laboratories.

78. The Observer from ISO informed the Committee that 37 experts representing 25 member countries were working in Working Group No. 7 (ISO/TC 34/WG 7 "Genetically modified organisms and derived products") in close cooperation with Working Group 11 of CEN/TC 275 on seven different items of GMO analysis and indicated that there was a good collaboration between the participating countries world-wide.

79. Many delegations and Observers pointed out the complexity of issues involved in GM analysis and supported further work on these issues.

80. The Committee concluded that the criteria approach should be applied in the selection of analysis for foods containing genetically modified material.

81. The Committee agreed that a Working Group led by Germany and the United Kingdom would update and further develop the paper prepared for this session and prepare recommendations for quality control measures in laboratories and criteria for method of analysis for consideration by the next Session of the Committee. The following countries and international organizations expressed their willingness to participate in this work: Argentina, Australia, Brazil, Canada, Egypt, France, Iran, Ireland, Italy, Japan, Malaysia, Netherlands, Philippines, United States, European Commission, AOAC, AOCS, EUROPABIO, ISO.

CONSIDERATION OF METHODS FOR THE DETECTION AND IDENTIFICATION OF FOODS DERIVED FROM BIOTECHNOLOGY: METHODS SUBMITTED BY THE *AD HOC* INTERGOVERNMENTAL TASK FORCE ON FOODS DERIVED FROM BIOTECHNOLOGY (Agenda Item 7 a)[14]

82. The Committee recalled that the Delegation of Germany had compiled the List of Methods prepared in response to CL 2001/18-FBT and that the Third Session of the *Ad Hoc* Intergovernmental Task Force on Foods Derived from Biotechnology forwarded the agreed List for consideration to the CCMAS. The Committee expressed it appreciation to the Task Force and to the Delegation of Germany for their considerable work.

83. The Delegation of Germany stressed the importance of this List as a reference that would be helpful for Member Governments. The Delegation suggested that this List should be considered by the *Ad Hoc* Working Group established earlier (see para. 81 above).

84. The Delegation of the Netherlands, supported by Germany, proposed that this List could be forwarded to the Committee on Food Labelling in order to facilitate the establishment of provisions on the labelling of GMOs.

[14] CX/MAS 02/8

85. The Delegation of the United States was of the view that methods should not be selected in the absence of specific provisions and that it was the responsibility of the Committee on Food Labelling to establish labelling requirements.

86. The Committee noted that the List provided a very good review of methods currently used by Member Governments in the area of GM material analysis and was available in document CX/MAS 02/8 for reference. However the Committee agreed that the selection or endorsement of methods without appropriate provisions was not possible. It also recalled its earlier decision to focus on the criteria approach (see para. 81) and agreed to inform the Committee on Food Labelling accordingly.

SINGLE LABORATORY VALIDATION: CONSIDERATION OF HARMONIZED IUPAC GUIDELINES FOR THE IN-HOUSE VALIDATION OF METHODS OF ANALYSIS (Agenda Item 8a)[15]

87. The Committee recalled that single-laboratory validation was under consideration for Codex purposes especially in the area of residue analysis and that it had agreed at its last Session to consider the published version of the IUPAC *Harmonized Guidelines for the In-House Validation of Methods of Analysis* with a view to adopting them by reference. It also recalled that the final version of the recently published above Guidelines had been circulated for comments.

88. The Delegation of the United Kingdom drew the attention of the Committee to the fact that the IUPAC *Harmonized Guidelines for Single-Laboratory Validation of Methods of Analysis* were not intended to be a detailed protocol but rather an overarching document. It included references to protocols for single laboratory method validation and could be used for Codex purposes. The Delegation indicated that this matter had been considered at the 15th IAM, that a number of new developments were coming forward from the EU, NMKL, IAEA and IUPAC and that the IAM would prepare a paper on available documents on single-laboratory validation.

89. Many delegations supported the proposal of the Delegation of the United Kingdom to adopt the IUPAC Guidelines for Codex purposes by reference.

90. The Delegation of Ireland drew the attention of the Committee to the EURACHEM Guide *"Fitness for purpose of analytical methods"* that could provide useful guidance for food laboratories on single-laboratory validation.

91. The Delegation of Japan while not opposing the adoption of the above Guideline by reference, drew the attention of the Committee to the fact that there were differences in definitions between the Guidelines and the current definitions in the Codex Procedural Manual, therefore it was necessary to ensure coherence and accuracy across these texts and the definitions presented in the Procedural Manual should take precedence. Some delegations indicated that terminology was not completely harmonized between various international organizations. It was pointed out that the definitions in the Manual might require updating to take into account the developments related to analytical terminology at the international level. This view was supported by some delegations.

92. The Delegation of Czech Republic indicated that in the past there had been cases when texts adopted by reference had slightly different definitions, therefore proposed to revise and up-date the definitions of the Procedural Manual.

93. After some discussion, the Committee agreed to include a footnote to the effect that the definitions applied only for the purposes of the Guidelines and were not generally applicable for Codex purposes.

94. The Committee agreed that the preferred approach should always be collaborative studies and only where it was not possible suggested to use single-laboratory validation.

95. The Committee agreed to recommend to the 26th Session of the Commission to adopt the IUPAC *Harmonized Guidelines for Single-Laboratory Validation of Methods of Analysis* by reference for Codex purposes (see Appendix III). It also agreed to initiate the revision of the definitions contained in the Codex Procedural Manual (Analytical Terminology for Codex Use), subject to the approval of the Commission as new work. A circular letter asking for comments on the current definitions would be sent for this purpose.

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15 CX/MAS 02/10; CX/MAS 02/10-Add.1(comments of the United States); CRD 12 (comments of Brazil, Czech Republic); CRD 13 (comments of the Philippines)

REQUIREMENTS FOR SINGLE-LABORATORY VALIDATION FOR CODEX PURPOSES (Agenda Item 8b)[16]

96. The Committee recalled that its last session had considered the use of single-laboratory validation for the purposes of Codex, taking into account the activities of international organizations and the work underway in other Codex Committees, and had agreed that the Delegation of the Netherlands would further develop general requirements for single-laboratory validation for Codex purposes.

97. The Delegation of the Netherlands stressed the importance of single-laboratory validation in the field of residue analysis and in order to address new hazards, and proposed to include in the Procedural Manual criteria for single-laboratory validated methods. The Committee considered the proposed *General Criteria* and made the following amendments.

98. The Committee noted that he IUPAC Guidelines were not a protocol but included reference to international protocols and amended the text accordingly.

99. The Committee agreed that the single-laboratory validated method should be embedded in a "quality system" rather than a "quality assurance system". After some discussion, it was also agreed to delete the reference to accreditation and to specify that the system should comply with ISO/IEC 17025.

100. The Delegation of Germany expressed the view that interlaboratory reference could be provided by three methods : a) calibration using reference materials; b) comparison of results achieved with other methods and; c) systematic participation in proficiency testing. The Delegation of the Netherlands stressed the importance of proficiency testing schemes, that should be considered as a priority to provide external reference. After an exchange of views, the Committee agreed to delete the third indent of the Criteria that listed these three options.

101. As a result of these discussions, the Committee agreed that the following text would be acceptable:

General Criteria for the Acceptance of Single-Laboratory Validated Methods of Analysis

Especially in the case of multi-analyte-multi-substrate methods and new hazards, interlaboratory validated methods may not be available or appropriate. Criteria used to select a method include the General Criteria for the Selection of Methods of Analysis, where appropriate. In addition, the single-laboratory validated methods must fulfil the following criteria:

i the method is validated according to an internationally recognized protocol (e.g. those referenced in the *Harmonized IUPAC Guidelines for the Single-Laboratory Validation of Methods of Analysis*)

ii the single-laboratory validated method is embedded in a quality system complying with ISO/IEC 17025

102. However, the Committee could not agree on the modalities of its incorporation into the Procedural Manual. It was recalled that these *General Criteria* had been proposed for inclusion after the *General Criteria for the Selection of Methods of Analysis using the Criteria Approach* and had not been associated with a specific Type of method in earlier discussions. However, some delegations expressed the view that these recommendations could not be included in the Manual as General Criteria, but should be restricted to Type IV methods because Type II and III methods should be collaboratively tested.

103. Other delegations recalled that the purpose of single-laboratory validation was to allow the use of reference methods that would not otherwise be available and that the current requirements for the type of methods would have to be amended accordingly. It was also pointed out that there was no need to apply additional requirements to Type IV methods and that the inclusion of criteria for single-laboratory validation was not relevant if they were not generally applicable.

104. The Committee could not come to a conclusion on an amendment to the Procedural Manual and agreed to inform the Committee on Pesticide Residues, the Committee on Residues of Veterinary Drugs in Foods and the Committee on Food Additives and Contaminants of the above discussion as the use of single-laboratory validation was especially important for their work.

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16 CX/MAS 02/11

SINGLE LABORATORY VALIDATION: VALIDATION OF METHODS THROUGH THE USE OF RESULTS FROM PROFICIENCY TESTING SCHEMES (Agenda Item 8 c)[17]

105. The Committee recalled that at the last session it had agreed to consider a paper on the validation of methods of analysis through the results from proficiency testing schemes. The Delegation of the United Kingdom introduced the paper and pointed out that in some situations there was a possibility of validating methods if there were enough participants in the proficiency testing scheme that used the same defined method of analysis or if a method was prescribed by co-ordinators. The Delegation indicated that this approach was more applicable in the areas of microbiological and GM analysis and that the annexes of the document provided practical examples on the validation of the method for the enumeration of *Listeria monocytogenes* in meat and meat products and on statistical analysis of the results from an ongoing proficiency testing scheme.

106. The Delegation drew the attention of the Committee to the fact that the International Harmonized Protocol for Proficiency Testing of (Chemical) Analytical Laboratories would be revised in the near future, therefore proposed that it might be useful if during the revision the harmonized protocol addressed the issue of method validation through the use of proficiency test results.

107. The Delegation of Poland pointed out the usefulness of the document and that the revised protocol would give guidance in terms of method validation. Many delegations supported this view especially if it would provide guidance in the design of proficiency testing schemes.

108. The Committee thanked the Delegation of the United Kingdom for their valuable document and supported the recommendations of the paper. It agreed to encourage IUPAC to work in this area.

THE USE OF ANALYTICAL RESULTS: SAMPLING, RELATIONSHIP BETWEEN THE ANALYTICAL RESULTS, THE MEASUREMENT UNCERTAINTY, RECOVERY FACTORS AND THE PROVISIONS IN CODEX STANDARDS (Agenda Item 9)[18]

109. The Committee recalled at its last Session it had noted that there were number of decisions that might be taken by those responsible for the enforcement of Codex analytical provisions which directly affected decisions as to whether a lot was in compliance with Codex requirements and therefore it was proposed that a paper be prepared for consideration of the issues involved.

110. The Delegation of the United Kingdom introduced the document and indicated that decisions regarding the acceptability of a lot or sample should be based on a concept that takes sampling and analytical aspects into consideration. The Delegation pointed out that at the present time there was no common understanding and interpretation of analytical results among Codex Members and therefore different decisions might be taken after an analysis of the same sample. The Delegation indicated that it occurred because some countries took into account uncertainty for the interpretation of results while others did not and that different sampling regimes were used. The Delegation indicated that approaches to solve these problems were presented in the annexes of the document. The Delegation proposed that when Commodity Committees develop specifications they should do it with respect to those factors which affect the interpretation of specifications. Therefore Commodity Committees should give clear guidance to the Committee on Methods of Analysis and Sampling on how they wished Codex specifications to be enforced.

111. Many delegations emphasized the importance of this issue in order to ensure consistency throughout Codex and supported efforts in this field.

112. The Observer from the EC pointed out that this matter was of major importance as issues related to the correction for measurement uncertainty, recovery and sampling uncertainty had consequences which could not be ignored. The Observer informed the Committee about the ongoing work in the EC and indicated that a draft document in this area was available for information.

113. The Delegation of Germany indicated that from the scientific point of view there was a need to take into account uncertainty and that was consistent with the requirement to prove "beyond reasonable doubt" that a limit had been exceeded. The Delegation pointed out that there should be a mechanism to ensure that

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17 CX/MAS 02/12; CRD 13 (comments of the Philippines)

18 CX/MAS 02/13

commodities were dealt with consistently and proposed to work on general recommendations for Commodity Committees.

114. The Delegation of the Netherlands drew the attention of the Committee to the fact that in the area of pesticide residues there was no correction for recovery while in other areas correction was applied and it was not clear enough to whom recommendations could be addressed. The Delegation therefore suggested to make relevant changes in the Procedural Manual so as to ensure that Commodity Committees applied a consistent approach on this issue.

115. The Delegation of Ireland indicated that this matter had been linked with dispute situations related to analytical and sampling errors and suggested to proceed in a step-wise manner by drawing very pragmatic guidelines. The Delegation informed the Committee that the International Laboratory Accreditation Body had established guidelines in this regard which were available from their website (www.ilac.org).

116. Some delegations were of the view that before proceeding further this problem should be addressed by Commodity Committees as they should consider how the analytical results would be used when developing provisions in Codex Standards.

117. The Committee agreed to forward this document containing explanatory notes (CX/MAS 02/12) to Commodity Committees for their consideration and comments. The Committee also agreed to forward this document to the Committee on Food Import and Export Inspection and Certification Systems and ask its advice insofar as inspection issues were involved.

REPORT OF AN INTER-AGENCY MEETING ON METHODS OF ANALYSIS (Agenda item 10)[19]

118. The Chairman of IAM (Dr. Roger Wood) introduced the draft report of the 15th Interagency Meeting and informed the Committee that several issues related to the work of the Committee had been discussed such as IAM membership, Criteria Approach, Single-Laboratory Validation, Electronic Compendium of Analytical Methods (e-CAM), Proficiency Testing and Harmonization of Analytical Terminology etc.

119. Dr. Wood noted that an electronic compendium of analytical methods (e-CAM) was introduced as an AOAC project. It was indicated that the system would provide summary information that would be useful when the criteria approach was adopted in Codex. It was also noted that e-CAM would be re-drafted by AOAC and would be accessible by all members of IAM.

120. Dr. Wood also indicated that it might be possible to give information on whether specific methods of analysis may be validated through proficiency testing scheme results if sufficient participants used a defined method. In this regard, he noted that IUPAC would consider the revision of the International Harmonised Proficiency Testing Protocol.

121. The Delegation of France supported by other delegations and observers asked for clarification regarding the IAM membership in view of its Terms of Reference. The Committee noted that the review of the IAM membership was an internal matter of IAM.

122. As regards the questions raised in the IAM report on the update of references for methods in Codex publications, the Secretariat indicated that only the amendments proposed by Codex Committees, endorsed by the Committee on Methods of Analysis and Sampling and adopted by the Commission could be included in revised Codex publications after each session of the Commission. Any proposal for update of methods would have to be put forward in the relevant Codex Committee. It was also recalled that member countries and international organizations had the opportunity to provide comments on the endorsement of methods of analysis for consideration by the Commission.

123. The Committee expressed its appreciation to the IAM for their constructive work and contribution to the work of the Committee and noted that the final IAM report would be placed on the AOAC website at: http://www.aoac.org/

OTHER BUSINESS AND FUTURE WORK (Agenda item 11)

124. The Committee noted that, as a result of the discussions at the current session, the next session would consider the following items:

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[19] CRD 2 (Report of the 15th Meeting of international organizations working in the field of methods of analysis and sampling (Inter-Agency Meeting)

- Draft General Guidelines on Sampling
- Draft Guidelines on Measurement Uncertainty
- Proposed Draft Guidelines for Evaluating Acceptable Methods of Analysis
- Proposed Draft Guidelines for Settling Disputes over Analytical (Test) Results
- Criteria for the Methods for foods derived from biotechnology
- Methods of Analysis for the determination of dioxins
- Endorsement of Methods in Codex Standards
- Review of current definitions in the Procedural Manual
- Consideration of the Use of Analytical results

DATE AND PLACE OF THE NEXT SESSION (Agenda item 12)

125. The Committee was informed that the 25th Session of the Codex Committee on Methods of Analysis and Sampling was tentatively scheduled to be held in Budapest in the spring of 2004. The exact date and place would be determined between the host country and the Codex Secretariat. The Committee was also informed that the Committee would be heldon an annual basis after its 25th Session.

SUMMARY STATUS OF WORK

Subject Matter	**Step**	**Action by**	**Document Reference in ALINORM 03/23**
Proposed amendments to the Procedural Manual: – Amendment to the *General Criteria for the Selection of Methods of Analysis Using the Criteria Approach* – new section on *Working Instructions for the Implementation of the Criteria Approach in Codex*		CCGP Governments 26th CAC	para. 42 Appendix II
IUPAC *Guidelines for Single-Laboratory Validation of Methods of Analysis* (for adoption by reference)	(*)	Governments 26th CAC	para. 55 Appendix III
Endorsement of methods of analysis, including general methods		Governments 26th CAC	paras. 57-70 Appendix VI
Proposed Draft General Guidelines on Sampling	5	Governments 26th CAC 25th CCMAS	para. 19 Appendix IV
Proposed Draft Guidelines on Measurement Uncertainty	5	Governments 26th CAC 25th CCMAS	para. 52 Appendix V
Proposed Draft Guidelines for evaluating acceptable methods of analysis	3	Governments 25th CCMAS	para. 34 Appendix VII
Proposed Draft Guidelines for Settling Disputes on Analytical (Test) Results	1/2/3	26th CAC France/Governments 25th CCMAS	para. 32
Review of Analytical Terminology for Codex Use (Procedural Manual)		26th CAC/Governments 25th CCMAS	para. 95
Criteria for methods of analysis for foods derived from biotechnology		Germany/United Kingdom/Governments 25th CMAS	para. 81
Use of Analytical Results		CCFICS Commodity Committees 25th CCMAS	para. 117
Methods for dioxins and PCBs		Germany/Governments 25th CCMAS	para. 5

(*) Equivalent to Step 8

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PROPOSED AMENDMENTS TO THE PROCEDURAL MANUAL

1. AMENDMENT TO THE *GENERAL CRITERIA FOR THE SELECTION OF METHODS OF ANALYSIS USING THE CRITERIA APPROACH*

In the case of Codex **Type II and** Type III methods, method criteria may be identified and values quantified for incorporation into the appropriate Codex commodity standard. Method criteria which are developed will include the criteria in section Methods of Analysis, paragraph (c) above together with other appropriate criteria, e.g., recovery factors."

2. *WORKING INSTRUCTIONS FOR THE IMPLEMENTATION OF THE CRITERIA APPROACH IN CODEX*

(for inclusion at the end of the *Principles for the Establishment of Codex Methods of Analysis* after the above *General Criteria*)

Any Codex Commodity Committee may continue to propose an appropriate method of analysis for determining the chemical entity, or develop a set of criteria to which a method used for the determination must comply. In some cases a Codex Commodity Committee may find it easier to recommend a specific method and request the Codex Committee on Methods of Analysis and Sampling (CCMAS) to "convert" that method into appropriate criteria. The Criteria will then be considered by the CCMAS for endorsement and will, after the endorsement, form part of the commodity standard replacing the recommended method of analysis. If a Codex Commodity Committee wishes to develop the criteria by itself rather than allowing the CCMAS to do so, it should follow instructions given for the development of specific criteria as outlined below. These criteria must be approved for the determination in question.

However, the primary responsibility for supplying methods of analysis and criteria resides with the Commodity Committee. If the Commodity Committee fails to provide a method of analysis or criteria despite numerous requests, then the CCMAS may supply an appropriate method and "convert" that method into appropriate criteria.

The minimum "approved" Codex analytical characteristics will include the following numeric criteria as well as the general criteria for methods laid down in the Analytical Terminology for Codex Use (see page 66):

- precision (within and between laboratories, but generated from collaborative trial data rather than measurement uncertainty considerations)
- recovery
- selectivity (interference effects etc.)
- applicability (matrix, concentration range and preference given to 'general' methods)
- detection/determination limits if appropriate for the determination being considered
- linearity

CCMAS will generate the data corresponding to the above criteria.

Conversion of Specific Methods of Analysis to Method Criteria by the CCMAS

When a Codex Commodity Committee submits a Type II or Type III method to CCMAS for endorsement, it should also submit information on the criteria listed below to enable the CCMAS to convert it into suitable generalized analytical characteristics:

- accuracy
- applicability (matrix, concentration range and preference given to 'general' methods)
- detection limit
- determination limit

- precision; repeatability intra-laboratory (within laboratory), reproducibility inter-laboratory (within laboratory and between laboratories), but generated from collaborative trial data rather than measurement uncertainty considerations
- recovery
- selectivity
- sensitivity
- linearity

These terms are defined in the Analytical Terminology for Codex Use (see page 66), as are other terms of importance.

The CCMAS will assess the actual analytical performance of the method which has been determined in its validation. This will take account of the appropriate precision characteristics obtained in collaborative trials which may have been carried out on the method together with results from other development work carried out during the course of the method development. The set of criteria that are developed will form part of the report of the CCMAS and will be inserted in the appropriate Codex Commodity Standard.

In addition, the CCMAS will identify numeric values for the criteria for which it would wish such methods to comply.

Assessment of the Acceptability of the Precision Characteristics of a Method of Analysis

The calculated repeatability and reproducibility values can be compared with existing methods and a comparison made. If these are satisfactory then the method can used as a validated method. If there is no method with which to compare the precision parameters then theoretical repeatability and reproducibility values can be calculated from the Horwitz equation. (M. Thompson, *Analyst*, 2000, **125**, 385-386).

Additions to ANALYTICAL TERMINOLOGY FOR CODEX USE [1]

Terms to Be Used in the Criteria Approach

Detection Limit

The detection limit is conventionally defined as field blank + 3σ, where σ is the standard deviation of the field blank value signal (IUPAC definition).

However, an alternative definition which overcomes most of the objections to the above approach (i.e. the high variability at the limit of measurement can never be overcome) is to base it on the rounded value of the reproducibility relative standard deviation when it goes out of control (where 3 σ_R = 100%; σ_R = 33%, rounded to 50% because of the high variability). Such a value is directly related to the analyte and to the measurement system and is not based on the local measurement system.

Determination limit

As for detection limit except that 6σ or 10σ is required rather than 3σ.

However, an alternative definition that corresponds to that proposed for the detection limit is to use σ_R = 25%. This value does not differ much from that assigned to the detection limit because the upper limit of the detection limit merges indistinguishably into the lower limit of the determination limit.

Recovery

Proportion of the amount of analyte present or added to the test material which is extracted and presented for measurement.

[1] These Definitions are proposed on an interim basis: they are subject to modifation as a result of further harmonization.

Selectivity

Selectivity is the extent to which a method can determine particular analyte(s) in mixtures or matrices without interferences from other components.

Selectivity is the recommended term in analytical chemistry to express the extent to which a particular method can determine analyte(s) in the presence of interferences from other components. Selectivity can be graded. The use of the term specificity for the same concept is to be discouraged as this often leads to confusion.

Linearity

The ability of a method of analysis, within a certain range, to provide an instrumental response or results proportional to the quality of analyte to be determined in the laboratory sample. This proportionality is expressed by an a priori defined mathematical expression. The linearity limits are the experimental limits of concentrations between which a linear calibration model can be applied with a known confidence level (generally taken to be equal to 1%).

HARMONIZED IUPAC GUIDELINES FOR SINGLE-LABORATORY VALIDATION OF METHODS OF ANALYSIS

(Recommended to the Commission for adoption by reference)

The Harmonized IUPAC *Guidelines for Single-Laboratory Validation of Methods of Analysis* are recommended for adoption for Codex purposes by the 26th Session of the Commission with the following note to the title.

Note:

The definitions apply only for the purpose of the Guidelines and are not generally applicable for Codex purposes.

Reference

M. Thompson, S.L.R. Ellison and R. Wood. "Harmonized Guidelines For Single-Laboratory Validation Of Methods Of Analysis" *Pure Appl. Chem*., **74**, (5) 835 – 855 (2002)

PROPOSED DRAFT GENERAL GUIDELINES ON SAMPLING
(At Step 5 of the Procedure)

TABLE OF CONTENTS

PROPOSED DRAFT GENERAL GUIDELINES ON SAMPLING

PREAMBLE

RATIONALE

Codex Food Standards are aimed at protecting consumers' health and ensuring fair practices in the food trade.

Codex Methods of Sampling are designed to ensure that fair and valid sampling procedures are used when food is being tested for compliance with a particular Codex commodity standard. The sampling methods are intended for use as international methods designed to avoid or remove difficulties which may be created by diverging legal, administrative and technical approaches to sampling and by diverging interpretation of results of analysis in relation to lots or consignments of foods, in the light of the relevant provision(s) of the applicable Codex standard.

The present guidelines have been elaborated to facilitate the implementation of these goals by Codex Commodity Committees, governments and other users.

BASIC RECOMMENDATIONS FOR THE SELECTION OF CODEX SAMPLING PLANS

The present clause represents a pre-requisite to the use of these Guidelines, and is intended to facilitate the selection of Codex sampling plans, as well as to follow a systematic approach for this selection.

The following enumerates the essential points that the Codex commodity committees, Governments and other users should address for the selection of appropriate sampling plans, when setting-up specifications.[1]

1) **Existence (or not) of international reference documents** on sampling of the considered products
2) **Nature of the control**
   - Characteristic applicable to each individual item of the lot
   - Characteristic applicable to the whole lot (statistical approach)
3) **Nature of the characteristic to control**
   - Qualitative characteristic (characteristic measured on a pass/failed or similar basis, i.e. presence of a pathogen micro-organism)
   - Quantitative characteristic (characteristic measured on a continuous scale, for example a compositional characteristic)
4) **Choice of the quality level** (AQL or LQ)
   - In accordance with the principles laid down in the Codex Manual of Procedures and with the type of risk: critical/ non-critical non-conformities.
5) **Nature of the lot**
   - Bulk or pre-packed commodities
   - Size, homogeneity and distribution concerning the characteristic to control
6) **Composition of the sample**
   - Sample composed of a single sampling unit
   - Sample composed of more than one unit (including the composite sample)

[1] See also "Principles for the establishment or selection of Codex Sampling procedures : general instructions for the selection of methods of sampling", in the Codex Alimentarius Manual of Procedures.

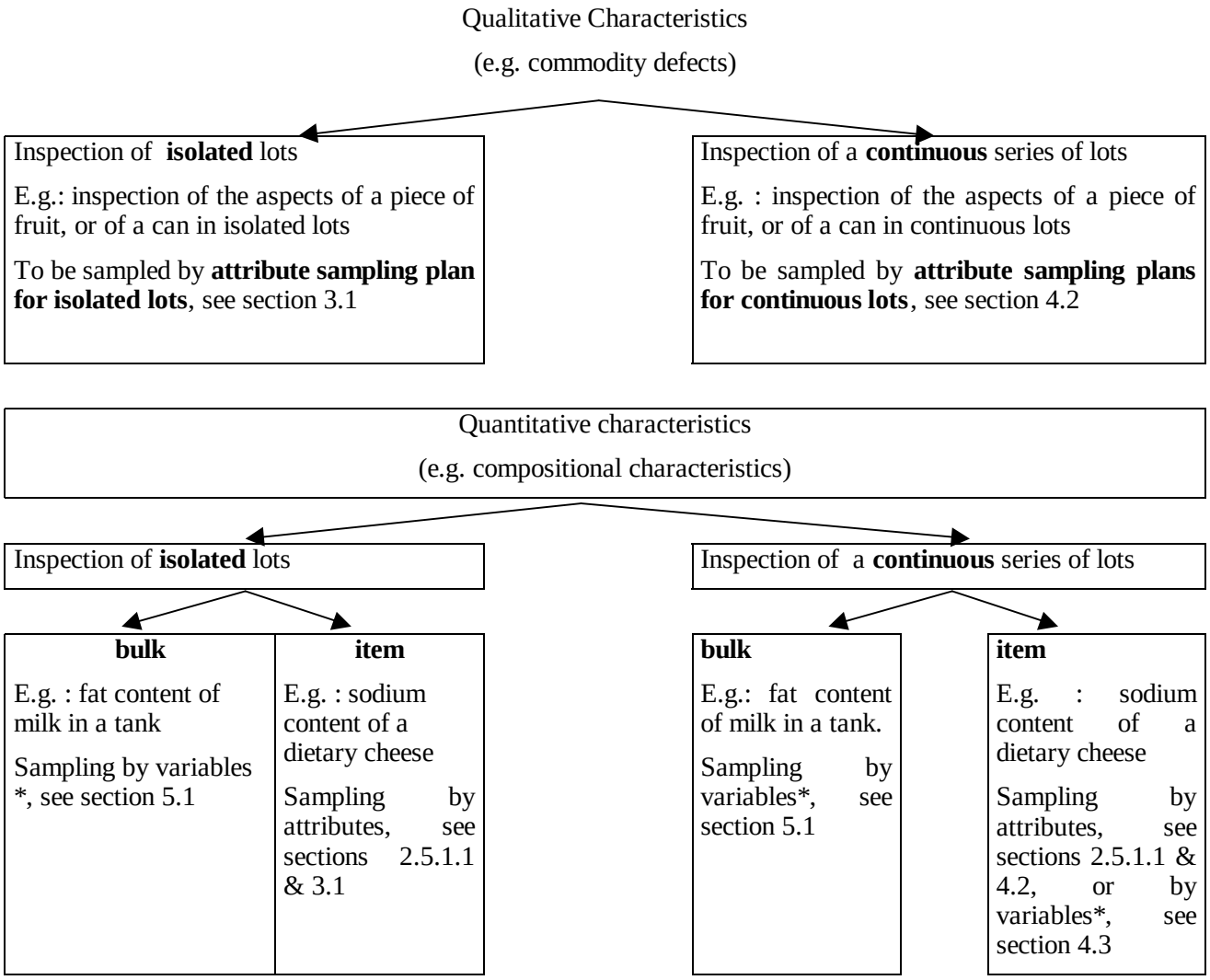
7) Choice of the type of sampling plan

- acceptance sampling plans for statistical quality control
  - for the control of the *average* of the characteristic
  - for the control of *per-cent non-conforming* items in the lot
    - Definition and enumeration of non-conforming items in the sample (**attribute** plans)
    - Comparison of the mean value of the items forming the sample with regards to an algebraic formula (**variable** plans).
- Convenience (or pragmatic, empirical) sampling plans[2]

The two flow-charts in the following pages sum up a systematic approach for the selection of a sampling plan and reference to the appropriate sections in the document, which does not cover sampling of heterogeneous bulk lots.

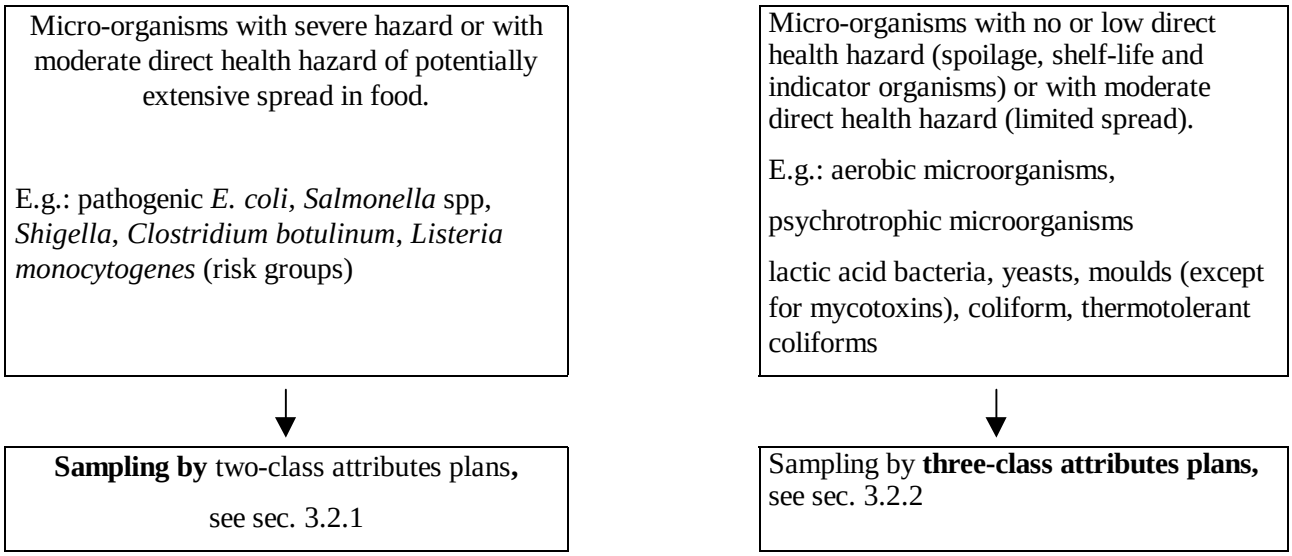
[2] Not covered by these Guidelines. Such pragmatic sampling has been used in the Codex for example for the determination of compliance with Maximum Residue Limits for pesticides and veterinary drugs.

FLOW-CHART FOR CHEMICAL AND PHYSICAL CHARACTERERISTICS



* normal distribution is assumed

FLOW-CHART FOR MICROBIOLOGICAL CHARACTERISTICS



8) Decision rules for the lot acceptance/rejection

See the appropriate references in Sections 3, 4 or 5.

SECTION I. PURPOSE OF CODEX GUIDELINES ON SAMPLING

1.1 PURPOSE

Sampling plans are required which ensure that fair and valid procedures are used when food is being controlled for compliance with a particular Codex commodity standard.

Since numerous, yet often complex, sampling plans are available it is the purpose of these guidelines to help those responsible for sampling to select sampling plans that are appropriate for statistical inspections under specifications laid down by Codex standards.

No sampling plan can ensure that every item in a lot conforms. These sampling plans are nevertheless useful for guaranteeing an acceptable quality level.

These guidelines contain the elementary principles of statistical control at reception, which complete the basic recommendations laid down in the Preamble.

1.2 TARGET AUDIENCE OF THE GUIDELINES

These Guidelines are above all aimed at Codex Commodity Committees which select from the plans recommended in sections 3, 4, and 5 those which at the time of the drafting of a commodity standard appear to them best suited for the inspection to be made. These Guidelines can also be used, if applicable, by governments in case of international trade disputes.

The Codex commodity committees, Governments and other users should be provided with the competent technical experts needed for good use of these guidelines, including the selection of appropriate sampling plans.

1.3 USERS OF SAMPLING PLANS RECOMMENDED BY THE GUIDELINES

The sampling plans described in these Guidelines may be implemented either by Governmental food control authorities, or by professionals themselves (self-inspection performed by producers and/or traders). In the latter case, these Guidelines enable the governmental authorities to check the appropriateness of the sampling plans implemented by the professionals.

It is recommended that the different parties concerned with sampling come to an agreement on the implementation of the same sampling plan for the respective controls.

1.4 SCOPE OF THE GUIDELINES

These Guidelines define at first in Section 2 general notions on food sampling, applicable in any situations, and then in Sections 3 to 5 cover certain situations of statistical food control, for whose certain sampling plans have been selected.

The following sampling situations are covered: for the control of only homogeneous goods,

- control of percentage of defective items by attributes or by variables, for goods in bulk or in individual items,
- control of a mean content.

These Guidelines do not cover the control of :

- non-homogeneous goods;
- for homogeneous goods, the cases where measurement error is not negligible compared to sampling error, as well as the control of a qualitative characteristic in a bulk material and;
- they do not deal with double, multiple and sequential sampling plans, deemed too complex in the frame of these Guidelines.

Detailed sampling procedures do not lie within the scope of these general guidelines. If necessary, they should be established by the Codex commodity committees.

These Guidelines are applicable for control at reception, and may not be applicable for control of end-products and for process control during production.

The following Table 1 summarises the situations covered by these Codex Guidelines and those, which are excluded**.** It also gives, where applicable, useful international references for some of the situations not covered by these Codex Guidelines.

TABLE 1 : GUIDE TO SELECTION OF SAMPLING PLANS FOR HOMOGENEOUS LOTS[3]

	Lots consisting of individualisable <u>bulk material</u>	**Lots consisting of <u>individual</u>[4] <u>items</u>**		
	Quantitative Measurements	Qualitative Measurements[5]	Quantitative Measurements	
Isolated lots	**Inspection by Variables of Bulk Materials for Percentage Non-conforming -Section 5.1** Example: check tank of milk to for added water	**Inspection by Attributes for percentage non-conforming - Section 2.5.1.1** Example: inspection of pieces of fruit for defects **Microbiological inspection of product - Section 3.1, 3.2** Example: testing uncooked vegetables for mesophilic aerobic micro-organisms.(see ICMSF standards)	**Inspection by Variables for percentage non-conforming - Section 4.3.2 (s method)** Example: to check whether fat content of a skimmed milk powder complies with Codex limit	**Average Content – Sections 3.3 and 4.4** Example: to check that average weight of items in a lot complies with label declaration (see also ISO 2854-1976, 3494-1976)

[3] Assuming for quantitative measurements, that measurement error is negligible in relation to process variation (see Section 2.4)

[4] Or individualisable.

[5] Qualitative data includes quantitative data classified as attributes, for example with respect to a limit.

Continuous series of lots	**Inspection by Variables of Bulk Materials for Percentage Non-conforming - Section 5.1** Example: check a tank of milk for added water	**Inspection by Attributes for percentage non-conforming - Section 2.5.1.1** Example: inspection of pieces of fruit for defects **Microbiological inspection of product -Section 3.1, 3.2** Example: testing uncooked vegetables for mesophilic aerobic micro-organisms (see ICMSF)	**Inspection by Variables for percentage non-conforming - Section 4.3.3 (σ method)** Example: to check whether fat content of a skimmed milk powder complies with Codex limit	**Average Content -** **Sections 3.3 and 4.4** Example: to check sodium content of a dietary food does not exceed prescribed level (See also ISO 2854-1974, 3494-1976)

1.5 RELATIONSHIP OF THE GUIDELINES WITH THE ISO GENERAL STANDARDS

In the cases of control situations dealt with by this document, the sampling shall only follow the rules of the sampling plans of this document, even if this document refers to the following ISO Standards for the details of the scientific and statistical background.

In the cases of control situations not dealt with by this document, and if they are dealt with by a general ISO Standard (see below), the product Committee or the governments should refer to them, and define how to use them[6].

The ISO Standards are provided in the following:

- ISO 2854 : 1976(E) : Statistical interpretation of data – Techniques of estimation and tests relating to means and variances
- ISO 2859-0:1995(E): Sampling procedures for inspection by attributes - Part 0: Introduction to the ISO 2859 attribute sampling system
- ISO 2859-1:1999(E): Sampling procedures for inspection by attributes - Part 1: Sampling plans indexed by acceptable quality level (AQL) for lot-by-lot inspection
- ISO 2859-2-1985(E): Sampling procedures for inspection by attributes - Part 2: Sampling plans indexed by limiting quality (LQ) for isolated lot inspection
- ISO 3494:1976 : Statistical interpretation of data – Power of tests relating to means and variances
- ISO 3951:1989(E): Sampling procedures and charts for inspection by variables for percent nonconforming
- ISO 7002:1986 (E) : Agricultural food products - Layout for a standard method of sampling a lot,
- ISO 8423:1991(E): Sequential sampling plans for inspection by variables for percent nonconforming (known standard deviation)
- ISO 8422:1991(E): Sequential sampling plans for inspection by attributes
- ISO/TR 8550:1994(E)): Guide for the selection of an acceptance sampling system, scheme or plan for inspection of discrete items in lots
- ISO 10725:2000(E): Acceptance sampling plans and procedures for the inspection of bulk material
- ISO/FDIS 11 648-1 : Statistical aspects of sampling from bulk materials – Part 1 : General principles
- ISO/DIS 14 560 : Acceptance sampling procedures by attributes – Specified quality levels in non-conforming items per million

The standards listed above were valid at the time of publication of these guidelines. However, since all standards are subject to revision, parties to agreements based upon these guidelines should ensure that the most recent editions of the standards are always applied.

SECTION 2. MAIN NOTIONS OF SAMPLING

INTRODUCTION

2.1.1 Presentation of the section

This section presents:

- the rationale and the procedure to be followed before sampling a lot and selecting a sampling plan (section 2.1.2);
- the vocabulary and the main notions used in sampling (section 2.2), particularly the principle of the operating characteristic curve of a sampling plan (section 2.2.12) and the related notions of acceptable quality and the limiting quality level (section 2.2.14). These notions are essential for risk assessment prior to selecting a plan;

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[6] It is recommended that Codex product committees also refer to existing sectorial ISO Standards (today approximately 20), which are specific to certain types of foods.

- sampling techniques, which are methods to collect and form the sample to be analysed (section 2.3);
- the different types of errors associated to the sampling plan (section 2.4);
- the types of sampling plans which lay down the rule for reaching a decision on the basis of the results obtained on samples taken from the inspected lot, in other words the acceptance or refusal of the lot after inspection (section 2.5);
- the principle of the inspection by single sampling plans by attributes (section 2.5.1.1) and by single sampling plans by variables (section 2.5.1.2) of percent nonconforming is presented and illustrated by the corresponding and compared operating characteristic curves (section 2.5.1.3);
- the selection of an attributes plan or a variables plan is illustrated by a diagram of the decision to be taken in terms of the inspection situations encountered (section 2.5.1.4);
- a table summarises the comparative advantages and disadvantages of an attribute plan and a variable plan (section 2.5.1.5).

2.1.2 General

Most of sampling procedures involve the selection of a sample (or samples) from a lot, the inspection or analysis of the sample, and the classification of the lot (as 'acceptable' or 'not acceptable') based upon the result of the inspection or analysis of the sample.

An acceptance *sampling plan* is a set of rules by which a lot is to be inspected and classified. The plan will stipulate the number of items, to be randomly selected from the lot under inspection, which will comprise the sample. A sampling procedure which involves '*switching*' (see Section 2.2.16) from one sampling plan to another is referred to as a '*sampling scheme*'. A collection of sampling plans and sampling schemes constitutes a '*sampling system*'.

Before elaborating any sampling plan, or before the Codex Committee on Methods of Analysis and Sampling endorses any plan, the Commodity Committee should also indicate the following:

- The basis on which the criteria in the Codex Commodity standards have been drawn up, for example;
    - whether on the basis that *a specified high proportion of items* in a lot, should comply with the provision in the standard, or
    - whether the *average of a set of samples* extracted from a lot must comply and, if so, whether a minimum or maximum tolerance, as appropriate, is to be given
- Whether there is to be any differentiation in the relative importance of the criteria in the standards. If so, the appropriate statistical parameter to be applied to each criterion should be indicated

Instructions on the procedure for implementing the sampling plan should indicate the following:

- The measures necessary in order to ensure that the sample taken is *representative* of the consignment or of the lot. (If a consignment consists of several lots, samples should be collected that are representative of the individual lots.)
- The samples shall be taken randomly, since they are more likely to reflect the quality of the lot, however information from a sample .may still not be identical with that from the whole lot due to sampling error.
- The *size and number of individual items* forming the sample taken from the lot or consignment
- The procedures to be adopted for *collecting, handling* and *recording* the sample(s)

The following issues should also be addressed when selecting a sampling procedure, in addition to the foreword:

- The distribution of the characteristic(s) in the population to be sampled
- The cost of the sampling plan
- Risk assessment (see Sections 2.2.11 and 2.2.14): Inspection systems, incorporating appropriate sampling plans, and designed to ensure food safety should be operated on the basis of objective risk assessment appropriate to the circumstances. Whenever possible, the risk assessment methodology

employed should be consistent with internationally accepted approaches; and should be based on current available scientific evidence.

The precise definition of an acceptance sampling procedure will require the setting or selection of:

- The characteristic to be measured
- Lot size
- An attribute or variables plan
- The Limiting Quality (LQ) level, for isolated lots; or the AQL (Acceptable Quality Level), for a continuous series of lots
- The level of inspection
- The size of the sample
- The criteria for acceptance or rejection of the lot
- The procedures to be adopted in cases of dispute

2.2 COMMONLY USED TERMS AND NOTIONS

The definitions of sampling terms used in these guidelines are mostly those specified in ISO 7002.

Some of the more commonly used terms in acceptance sampling are described in this section.

2.2.1 Lot

A lot is a definite quantity of some commodity manufactured or produced under conditions, which are presumed uniform for the purpose of these Guidelines.

For the goods presumed heterogeneous, sampling can only be achieved on each homogeneous part of this heterogeneous lot. In that case, the final sample is called a stratified sample (see 2.3.3).

NOTE: A **continuous series of lots** is a series of lots produced, manufactured or commercialised on a continuous manner, under conditions presumed uniform. The inspection of a continuous series of lots can only be achieved at the production or processing stage.

2.2.2 Consignment

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

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

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

2.2.3 Sample (representative sample)

Set composed of one or several items (or a portion of matter) selected by different means in a population (or in an important quantity of matter). It is intended to provide information on a given characteristic of the studied population (or matter), and to form a basis for a decision concerning the population or the matter or the process, which has produced it.

A **representative sample** is a sample in which the characteristics of the lot from which it is drawn are maintained. It is in particular the case of a simple random sample where each of the items or increments of the lot has been given the same probability of entering the sample.

Note : Sections A.11 to A.17 of Annex A of the Standard ISO 7002 define the composite sample, the reference sample, the global sample, the test sample, the laboratory sample, the primary sample and the reduced sample.

2.2.4 Sampling

Procedure used to draw or constitute a sample.

Empirical or punctual sampling procedures are sampling procedures, which are not statistical-based procedures, that are used to make a decision on the inspected lot.

2.2.5 Total estimation error

In the estimation of a parameter, the total estimation error is the difference between the calculated value of the estimator and the true value of this parameter.

The total estimation error is due to:

- sampling error,
- measurement error,
- rounding-off of values or sub-division into classes,
- bias of the estimator,
- other errors.

2.2.6 Sampling error

Part of the total estimation error due to one or several of the following parameters:

- the heterogeneity of the inspected characteristics,
- the random nature of a sampling,
- the known and acceptable characteristics of the sampling plans.

2.2.7 Item or increment of individualisable goods

a) Individualisable goods : Goods which can be individualised as items (see b) or in increments (see c), for example :

- a pre-package,
- a flask or a spoon containing a quantity of goods determined by the sampling plan, and taken from a lot, for example :
  - a volume of milk or of wine stored in a tank,
  - a quantity of goods taken from a conveyor belt,…

b) Item: An actual or conventional object on which a set of observations may be made, and which is drawn to form a sample.

Note: The terms “individual” and “unit” are synonymous with “item”

c) Increment: Quantity of material drawn at one time from a larger quantity of material to form a sample.

2.2.8 Sampling plan

Planned procedure which enables one to choose, or draw separate samples from a lot, in order to get the information needed, such as a decision on compliance status of the lot.

More precisely, a sampling plan is a scheme defining the number of items to collect and the number of non-confirming items required in a sample to evaluate the compliance status of a lot.

2.2.9 The Characteristic

A characteristic *is a property, which helps to identify, or differentiate between, items within a given lot* . The characteristic may be either quantitative (a specific measured amount, plan by variables) or qualitative (meets or does not meet a specification, plan by attributes). Three types of characteristic and associated types of sampling plan are illustrated in Table 2.

Table 2 : Sampling plans to be associated with the type of characteristic

Type of Characteristic	*Type of Sampling Plan*
Commodity defects : characteristics that may be expressed by two excluding situations as passed/not passed, yes/not, integer/not integer, spoiled/not spoiled (e.g. as applied to visual defects such as loss of colour, mis-grading, extraneous matter etc)	**'**Attributes' (e.g. as in Codex Sampling Plans for Pre-packaged Foods, CAC/RM 42-1969[7])
Compositional characteristics: characteristics that may be expressed by continuous variables. They may be normally distributed (e.g. most analytically determined compositional characteristics such as moisture content) or they may be non-normally distributed.	'Variables with unknown standard deviation' for normally distributed characteristics and 'attributes' for characteristics whose distributions deviate significantly from normal
Health-related properties (e.g. in the assessment of microbial spoilage, microbial hazards, irregularly occurring chemical contaminants etc)	Specified sampling plans to be proposed appropriate to each individual situation (e.g. for microbiological control, see Section 3.2). Plans to determine incidence rates in a population may be used.

2.2.10 Homogeneity

A lot is **homogenous** *relative to a given characteristic* if the characteristic is uniformly distributed according to a given probability law throughout the lot[8].

NOTE: A lot being homogeneous for a given characteristic does not mean that the value of the characteristic is the same throughout the lot.

A lot is **heterogeneous** relative to a given characteristic if the characteristic is **not** uniformly distributed throughout the lot. Items in a lot may be homogenous on one characteristic whilst heterogeneous on another characteristic.

2.2.11 Defects (Nonconformities) and Critical Nonconformities

A *defect (nonconformity)* occurs within an item when one or more, *quality characteristic* does not meet its established quality specification. A *defective item* contains one or more defects.

Lot quality may be judged in terms of the acceptable *percentage of defective items* or the *maximum number of defects (nonconformities) per hundred items, in respect of any type of defects* (see also Section 2.2.7 for the definition of an item).

Most acceptance sampling involves the evaluation of *more than one quality characteristic*, which may differ in importance with respect to quality and/or economic considerations. Consequently, it is recommended that nonconformities be classified as follows, according to their degree of seriousness (see also Section 2.2.9 for the definition of a characteristic):

- **Class A:** Those nonconformities considered to be of the highest concern in terms of the quality and/or safety of the product
- **Class B:** Those nonconformities considered to be less important than the Class A nonconformities

2.2.12 Operating Characteristic Curve

For a given sampling plan, an **Operating Characteristic (OC) curve** describes the probability of acceptance of a lot as a function of its actual quality. It relates the rate of defective items in lots (x-axis) with the

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[7] The Codex Alimentarius Commission at its 22nd Session (June 1997) abolished the CAC/RM Numbering System.

[8] After checking, if necessary by an appropriate statistical test for comparison of 2 samples, i.e. a parametric test of a mean/variance of the characteristic (e.g. Aspin-Welch test) or a non parametric test of the characteristic for the proportions (e.g. Chi-square test or Kolmogorof-Smirnof test) (see references 2 , 3 and 4).

probability of accepting these lots at control (y-axis). Section 4.1 develops the principle of such a curve and illustrates it with an example.

2.2.13 Producers' risk and consumers' risk

Producers' risk (PR)

On the OC curve (see 2.2.12) of a sampling plan, the producers' risk corresponds to the probability to reject a lot having a proportion P_1 of defective items (generally low), fixed by the sampling plan. According to the producer, such a lot should not be rejected.

In other words, the PR is the probability to wrongly reject a lot.

Generally, the PR is expressed by a proportion noted P_{95} corresponding to the proportion of defective items in the lot accepted in 95 % of the cases (i.e. rejected in 5 % of the cases).

Consumers' risk (CR)

On the OC curve (see 2.2.12) of a sampling plan, the consumers' risk corresponds to the probability to accept a lot having a proportion P_2 of defective items (generally low), fixed by the sampling plan. According to the consumer, such a lot should be rejected.

In other words, it is the probability to wrongly accept a lot.

Generally, the CR is expressed by a proportion noted as P_{10} which corresponds to the proportion of defective items in the lot accepted in 10 % of the cases (i.e. rejected in 90 % of the cases).

Discrimination Distance (D)

The discrimination distance (D) is the absolute distance between the producers' risk (PR) and the consumers' risk (CR), and should be specified, taking into account the values of the population standard deviations of sampling and of measurements.

2.2.14 The Acceptable Quality Level (AQL) and Limiting Quality (LQ) Level

The inspection of a lot using either an attributes or variables sampling plan will allow a decision to be made on the quality of the lot.

*The **Acceptable Quality Level** (AQL) for a given sampling plan is the rate of non-conforming items at which a lot will be rejected with a low probability, usually 5 %.*

The **Acceptable Quality Level** (AQL) is used as an indexing criterion applied to *a continuous series of lots* which corresponds to a maximum rate of acceptable defective items in lots (or the maximum number of defective items per hundred items). This is a quality goal fixed by the profession. This does not mean that all the lots having a rate of defective items greater than the AQL will be rejected at the control, but this means that the higher the rate of defective items exceeds the AQL, the greater is the probability of rejection of a lot. For any given sample size, the lower the AQL, the greater the protection for the consumer against accepting lots with high defective rates, and the greater the requirement for the producer to conform with sufficiently high quality requirements. Any value for AQL should be realistic in practice and be economically viable. If necessary, the value of AQL should take into account safety aspects.

It should be recognised that the selection of a value for the AQL depends on the specific characteristic considered and of its relevance (economic or other) for the standard in its whole. A risk analysis may be undertaken to assess the possibility and severity of negative impacts on public health caused, for example, by the presence in food products of additives, contaminants, residues, toxins or pathogenic micro-organisms.

The characteristics which may be linked to critical defects (for example to sanitary risks) shall be associated with a low AQL (i.e. 0,1 % to 0,65 %) whereas the compositional characteristics such as the fat or water content, etc may be associated with a higher AQL (e.g., 2,5 % or 6,5 % are values often used for milk products). The AQL is used as an indexing device in the tables of the Standards ISO 2859-1, ISO 3951 and in some tables of ISO 8422 and ISO 8423 (see section 1).

The AQL is particular producers' risk, generally different from P95 (see 2.2.13).

The **Limiting Quality** (LQ) for a given sampling plan is the rate of non-conforming items at which a lot will be accepted with a low probability, usually 10 %.

The **Limiting Quality** (LQ) is applied when *a lot is considered in isolation*. It is a quality level (expressed, for example, as percentage nonconforming items in the lot) which corresponds to a specified and relatively low probability of acceptance of a lot having a rate of defective items of LQ. Generally, the LQ corresponds to the rate of defective items of lots accepted after control in 10 % of the cases. LQ is an indexing device used in ISO 2859-2 (where it is recommended that the LQ is set at least three times the desired AQL, in order to ensure that lots of acceptable quality have a reasonable probability of acceptance).

The LQ is generally very low when the plans aim at the control of food safety criteria. It is often higher when the plans aim at the control of quality criteria.

The LQ is a particular consumers' risk, it corresponds to P_{10} (see 2.2.13).

The users of sampling plans shall mandatory agree on the choice on the AQL or LQ of the plan used for the quality control of the lots.

For a given product, a single AQL (or LQ) should be allocated to each of the two classes of nonconformities specified in Section 2.2.11, a low AQL (e.g. 0,65 %) being allocated to Class A nonconformities (e.g. pesticide content in follow-up milk), and a higher AQL (e.g. 6,5%) being allocated to Class B nonconformities (e.g. protein content in follow-up milk).

Consequently, there is a separate sampling plan for each of the two AQLs (LQs), and a lot is accepted only if it is accepted by each of the plans. The same sample may be used for each class provided the evaluation is not destructive for more than one type of nonconformity. If two samples must be collected they can be taken simultaneously for practical reasons.

2.2.15 Responsible Authority

The **responsible authority** will be the official designated by the importing country; and will normally be responsible, for example, for setting the '*inspection level'* and for the introduction of *'switching rules'* (see 2.2.16).

2.2.16 Inspection Levels and Switching Rules

The **inspection level** *relates the sample size to the lot size and hence to the discrimination afforded between 'good' and 'poor' quality*. For example, Tables I and I-A of ISO 2859-1:1989 (E) and ISO 3951:1989 (E) respectively provide seven and five inspection levels. For a given AQL the lower the inspection level number the greater is the risk of accepting poor quality lots.

The inspection level should be set by the '*responsible authority*'. *Unless otherwise specified, the normal (II) inspection level shall be used*. Reduced (I) level or tightened (III) level should be used when less or more discrimination, respectively, is required. Level II affords less than double the sample size of Level I, Level III gives about one and a half times the sample size of Level II. The 'special' levels (S-1 to S-4) should be used where relatively small sample sizes are required and large sampling risks can and/or must be tolerated.

A sampling scheme involves 'switching' between normal, tightened and reduced inspection sampling plans. It is recommended that all Commodity Committees include switching rules in those sampling plans applied to a continuing series of lots.

Normal inspection is designed to protect the producer against having a high proportion of lots rejected when the quality of the product is better than the AQL. However, if two out of any five (or fewer) successive lots are not accepted, then tightened inspection must be introduced. On the other hand, if production quality is consistently better than the AQL, sampling costs may be reduced (at the discretion of the responsible authority) by the introduction of reduced-inspection sampling plans.

Switching rules for a continuous series of lots are described in detail in Sections 4.2.2.4 and 4.3.4.

2.2.17 Acceptance Number

For a given attributes sampling plan, the **acceptance number** *is the maximum number of nonconforming units, or the maximum number of nonconformities, allowed in the sample if the lot is to be accepted. Zero acceptance number plans* are described in Sections 2.5.2.

2.2.18 Lot Size and Sample Size

For internationally traded commodities, the lot size is usually specified in the shipping manifest. If a different lot size is to be used for sampling purposes, this should be clearly stipulated in the standard by the appropriate Commodity Committee.

There is no mathematical relationship between sample size (n) and lot size (N). Therefore, mathematically, there is no objection to take a sample of small size to inspect an homogeneous lot of large size. Nethertheless, the ratio f = n/N influences the sampling error when the lot size is small. Moreover, in an objective of consumer protection (in particular health), it is recommended, as illustrated in the following example, to choose samples of larger sizes when the lot sizes are large.

> Example : Inspection of the fat content in whole milk of 8500 items by attribute sampling plans at AQL of 2,5 %.
>
> Two different plans could bed used : plan 1 (n = 5, c = 0, LQ = 36,9 %) and plan 2 (n = 50, c = 3, LQ = 12,9 %).
>
> Given the LQ of plan 1, lots having a non-conforming rate of 36,9 % (that is 3136 non-conforming items) are accepted in 10 % of cases.
>
> Given the LQ of plan 2, lots having a non-conforming rate of 12,9 % (that is 1069 non-conforming items) are accepted in 10 % of cases.
>
> The choice of plan 2 enables the avoidance of the risk in 10 % of the cases in placing on the market (3136-1069) = 2067 non-conforming items.

When the ratio f = n/N (where n is the sample size and N is the lot size) is less than or equal to 10 %, and when the lots are assumed to be homogenous, it is the absolute sample size that is more important rather than its relationship to the size of the lot.

However, in order to reduce the risk of accepting large numbers of defective items, it is usual to increase the sample size as the lot size increases, especially when it is assumed that the lot is not homogenous.

With a large lot it is possible and economical to take a large sample whilst maintaining a large lot-to-sample ratio and, thereby, achieving better discrimination (between acceptable and unacceptable lots). Furthermore, for a given set of sampling efficiency criteria, the sample size will not increase as rapidly as the lot size and will not increase at all after a certain lot size. However, there are a number of reasons for limiting the lot size:

- the formation of larger lots may result in the inclusion of a widely varying quality
- the production or supply rate may be too low to permit the formation of large lots
- storage and handling practicalities may preclude large lots
- accessibility for drawing random samples may be difficult with large lots
- the economic consequence of non-acceptance of a large lot is large.

2.3 SAMPLING PROCEDURES

2.3.1 Employment of Authorised Sampling Officers

It is highly recommended that the sampling is performed by persons trained and authorised in the techniques of sample collection by the importing country.

2.3.2 Material to be Sampled

Each lot that is to be examined must be clearly defined. The appropriate Codex Commodity Committee should stipulate how a consignment should be handled in instances where no lot designation exists.

2.3.3 Representative sampling

The representative sampling is a procedure used for drawing or forming a representative sample[9].

The requirements of this clause shall be, if needed, completed by procedures (such as how to collect and to prepare a sample). These procedures shall be defined by the users, in particular the Codex Products Committees.

Random sampling involves the collection of *n* items from a lot of *N* items in such a way that all possible combinations of *n* items have the same probability of being collected. The randomness can be obtained by use of table of random number which can be generated by using computer software.

In order to avoid any dispute over the representativeness of the sample, a random sampling procedure should be chosen, whenever possible, alone, or in combination with other sampling techniques.

Assuming the items can be numbered or ordered, even virtually when it is not possible to have individual items (e.g., in the case of a tank of milk or of a silo of grains), the choice of the items or of the increments entering into the sample should be done as follows:

1. To number all the items or increments of the lot (true or virtual)
2. The numbers of the items or increments to be sampled are determined randomly using Table 3 of the Standard ISO 2859-0:1995 or any approved table of random numbers.

The collection of samples is to be performed in a random manner, whenever possible during the loading or unloading of the lot.

If the lot is heterogeneous, a random sample may not be representative of the lot. In such cases, stratified sampling may be a solution. Stratified sampling consists of dividing the lot into different strata or zones, each stratum being more homogenous than the original lot. Then a random sample is drawn from each of these strata, following specified instructions which may be drafted by the Codex product committees. Each stratum can then be inspected by random sampling which usually includes from 2 to 20 items or increments per sample. (see the sampling plans of ISO 2859-1 of letter-codes A to F at the inspection level II). But before sampling, it is necessary, where appropriate, to refer to the specific instructions of the Codex product committees.

When it is not possible to sample at random[10], for example in a very large store where the goods are badly tidied or when the production process includes a periodic phenomenon (e.g. a contaminant which is specifically located in a particular area of the silo or a regulator detuned every each k seconds, such as every k seconds the products packaged by this regulator have defaults), it is mandatory :

1. To avoid preferentially choosing items which are more easily accessible or which can be differentiated by a visible characteristic.
2. In the case of periodic phenomena, to avoid sampling every k seconds or every k^{th} package, or every k^{th} centimetres, to take an unit from every n^{th} palette, pre-package,…

2.3.4 Preparation of samples

2.3.4.1 Primary Samples

A **primary sample** is the ‘portion of product’ collected from a lot during the first stage of the sampling process, and will normally be in the form of an item (if collected from a lot of prepacked products) or of an increment (if collected from a bulk lot). (However, an ‘increment’ may be considered to be an ‘item’ if measurements are made on individual increments.) As far as is practicable, primary samples should be taken throughout the lot and departures from this requirement should be recorded. Sufficient primary samples of similar size should be collected to facilitate laboratory analysis. In the course of taking the primary samples (items or increments), and in all subsequent procedures, precautions must be taken to maintain sample

[9] See the definition of a representative sample in 2.2.3.

[10] The assessment of such a situation can be done, for a periodic phenomenon, by looking at the process control chart, for the storage conditions, or by obtaining information from storage managers, laboratories, professional organisations.

integrity (i.e., to avoid contamination of the samples or any other changes which would adversely affect the amount of residues or the analytical determinations, or make the laboratory sample not representative of the composite sample from the lot).

2.3.4.2 Composite Sample

When required by the sampling plan, a **composite sample** is produced by carefully mixing the primary samples (items) from a lot of *pre-packaged* products; or by carefully mixing the primary samples (increments) from a *bulk* (*not* pre-packaged) lot.

Except for economical reasons, this sampling technique is not to be recommended given the loss of information on sample-to-sample variation due to the combination of primary samples.

2.3.4.3 Final Sample

The *bulk or bulked sample* should, if possible, constitute the **final sample** and be submitted to the laboratory for analysis. If the bulk/bulked sample is too large, the final sample may be prepared from it by a suitable *method of reduction*. In this process, however, individual items must not be cut or divided.

National legislative needs may require that the final sample be subdivided into two or more portions for separate analysis. Each portion must be representative of the final sample.

2.3.5 Packaging and Transmission of Laboratory Samples

The sample finally submitted to the laboratory is described as the **laboratory sample** and will take the form of either the final sample or a representative portion of the final sample.

The laboratory sample should be kept in such a manner that the controlled characteristic is not modified (e.g., for microbiological controls, mandatory use of a sterile and cooled container). Moreover, the laboratory sample should be placed in a clean inert container offering adequate protection from external contamination and protection against damage to the sample in transit. The container should then be sealed in such a manner that unauthorised opening is detectable, and sent to the laboratory as soon as possible taking any necessary precautions against leakage or spoilage, e.g., frozen foods should be kept frozen and perishable samples should be kept cooled or frozen, as appropriate.

2.3.6 Sampling reports

Every sampling act implies the drafting of a sampling report as described in clause 4.16 of the Standard ISO 7002 and indicating in particular the reason for sampling, the origin of the sample, the sampling method and the date and place of sampling, together with any additional information likely to be of assistance to the analyst, such as transport time and conditions. The samples, in particular the ones for the laboratory, shall be clearly identified.

In case of any departure from the recommended sampling procedure (when it was necessary, for any reason, to deviate from the recommended procedure), it is necessary to append to the sampling report another detailed report on the deviating procedure which has been actually followed. However in this case, no decision can be taken at control, this decision is to be taken by the responsible authorities.

2.4 ESTIMATION ERRORS

Quantitative results are of only limited value if they are not accompanied by some estimate of the *random* (unpredictable) and *systematic (*predictable*)* errors in them. (*Random* errors affect the precision of the result, whereas *systematic* errors affect accuracy.).

Sampling plans are associated with two types of error:

- *sampling error* (caused by the sample failing to accurately represent the population from which it was collected); and
- *measurement error* (caused by the measured value of the characteristic failing to accurately represent the true value of the characteristic within the sample).

It is desirable that the sampling errors associated with any sampling plan, as well as the measurement errors associated with the analysis should be quantified and minimised.

- General case

Generally, it is assumed that analytical error is negligible compared to sampling error (e.g., analytical error is at most 1/3 of sampling error, then the standard deviation for the observed results will be less than 5 % than the standard-deviation without taking into account the analytical error [11]).

- First specific case : measurement error of the same order of magnitude than sampling error

When the controlled characteristics need to be analysed, any decision on a lot from a sample shall take into account the analytical error, in comparison with the sampling error if the latter is of the same order of magnitude. In such case, the standard-deviation for the observed results will be less than 41 % than the standard-deviation without taking into account the analytical error [12].

These Guidelines do not address how to take analytical error into consideration.

- Second specific case : measurement error larger than sampling error

In that case, there is no need to apply any statistical sampling plan.

2.5 TYPES OF SINGLE SAMPLING PLANS

2.5.1 Single sampling plans for inspections of percent non-conforming items

2.5.1.1 Principles of inspection by attributes of percent non-conforming items

The following text and curves present simply the principles of inspection by single sampling plans by attributes and by variables of percent nonconforming as well as their efficacy.

A sampling plan for inspection by attributes is a method for evaluating the quality of a lot which operates by classifying each increment of the sample as a conforming or nonconforming characteristic or attribute, depending on whether the Codex standard specification is complied with or not. This characteristic is either qualitative (for example the presence of a blemish on fruit) or quantitative (for example the sodium content of a dietary food, classified as conforming or non-conforming in relation to a limit noted). The number of increments having the nonconforming attribute are then counted and if the acceptance number set by the plan is not exceeded the lot is accepted, otherwise it is refused.

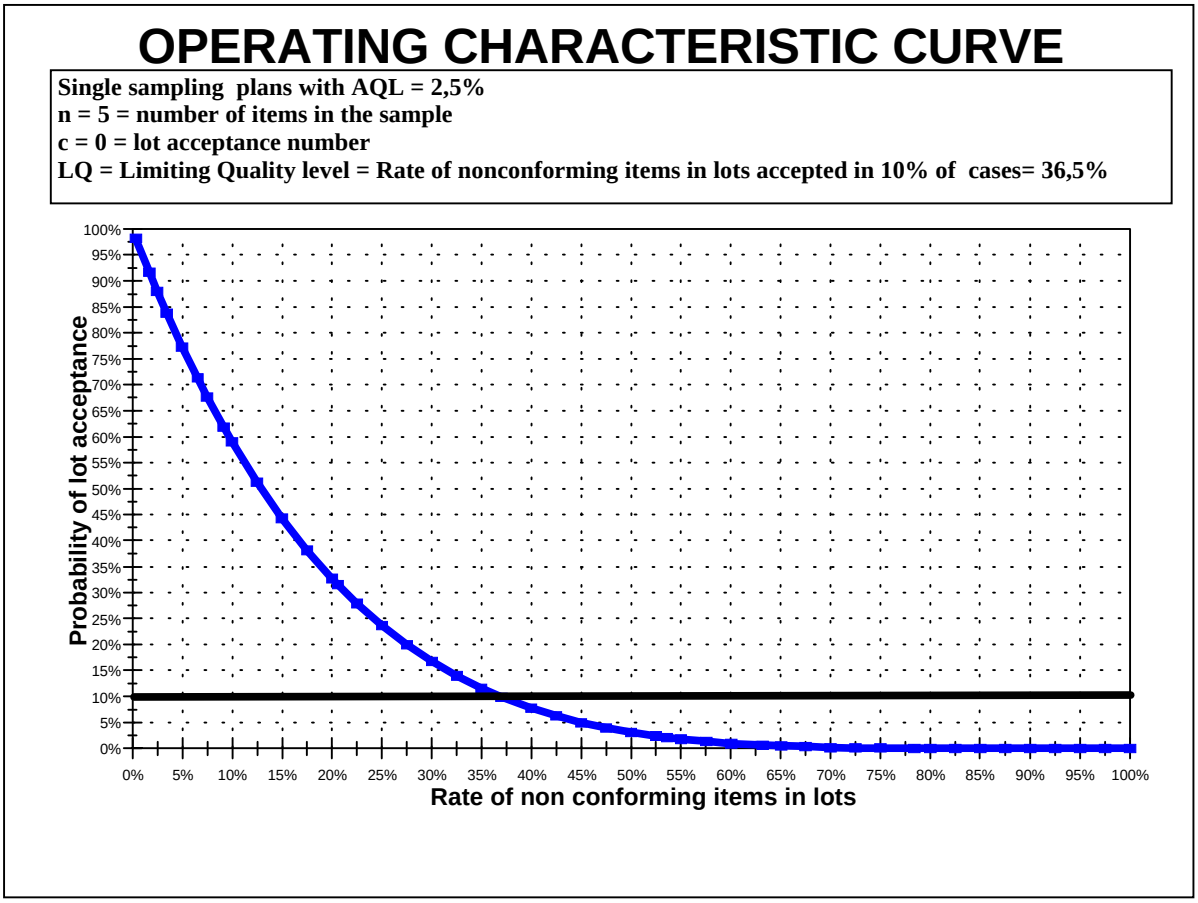
> EXAMPLE 1 : A single sampling plan by attributes of AQL = 2,5 % to inspect the sodium content of a lot of dietary cheese low in sodium for which the maximum sodium content is set by Codex standard 53-1981 at 120 milligrams per 100 grams of commodity (noted U = 120 mg/100 g).
>
> Decision to be taken according to this plan:
>
> The lot is accepted if there is no nonconforming increment (c = 0) in a sample of five increments (n = 5), a nonconforming increment being one whose sodium content -given the analytical tolerances- is higher than the specification relative to sodium in dietary cheeses, i.e. 120 milligrams.
>
> The following Figure 1 is the characteristic operating curve of this plan. It shows that in 50 % of the cases, lots having 13 % of defective items are accepted at inspection.

---

[11] The total standard-deviation $\sigma = \sqrt{\sigma_s^2 + \sigma_m^2}$, where σ_s is the sampling standard-deviation, σ_m the measurement Standard-deviation. If $\sigma_m = \sigma_s/3$, then $\sigma = \sqrt{\sigma_s^2 (1 + 1/9)} = 1{,}05 \times \sigma_s$, or an increase of 5 %.

[12] With the same notations than in the former foot-note, if $\sigma_s = \sigma_m$, then $\sigma = \sqrt{2 \times \sigma_s^2} = 1{,}41 \times \sigma_s$, or an increase of 41 %.

Figure 1: OC Curve, attribute sampling plan



EXAMPLE 2 : Single sampling plan by attributes, AQL = 6,5 %, for the inspection of the quality of pre-packed quick frozen peas.

Characteristics of the plan:

Criterion of non-conformity : the pre-packed bag contains more than 15 % m/m of defective peas (blond peas, blemished peas,…)

Number of sample units : n=13

AQL = 6,5 %

Acceptance number : c = 2 = maximum acceptable number of defective bags in the sample (acceptance criterion of the lot)

Rejection number : Re = 3 = minimum number of defective bags in the sample which implies the rejection of the lot (rejection criterion of the lot)

Decision to be taken according to this plan:

The lot is accepted if there is no more than 2 defective bags in a sample of 13 bags.

2.5.1.2 Principles of inspection by variables of percent noncon forming

2.5.1.2.1 General

A sampling plan by variables is a method for evaluating the quality of a lot which consists of measuring for each item the value of a variable characterising the inspected commodity.

EXAMPLES (To illustrate the difference between the attribute and variable sampling plans, the example for dietary cheese at maximum content of sodium is used for the variable plans)

- The maximum sodium content U of a dietary cheese low in sodium, for which the maximum sodium content is fixed by the Codex standard 53-1981 at 120 milligrams per 100 grams of product ;
- The minimum fat content L of a whole milk;
- A range of values, such as the vitamin A content of an infant formula, between L and U.

The inspection consists of measuring the variable characterising the inspected good for each of the *n* items forming the sample, then in calculating the mean value *x* of these *n* items in the sample.

The decision concerning acceptance or rejection of the lot is made by comparing this mean content *x* with the numeric value of an algebraic expression including :

- either U the maximum value of the specification (case of a maximum value to inspect), either L the minimum value of the specification (case of a minimum value to inspect), either L and U (case of a range of values to inspect) ;
- the standard deviation of the values of the variable inspected in the lot ;
- an acceptance constant K, determined by the sampling plan and depending on the AQL distribution law of the measured variable.

The algebraic expression depends also on the fact that the standard deviation is known or unknown. The decision formulae are given in 2.5.1.2.2 and 2.5.1.2.3.

2.5.1.2.2 The standard deviation σ of the distribution is known (σ-method)

The σ-method (see 2.2.19) is used for example in the case of inspections made by professionals who, owing to the large number of inspections they make, know the standard deviation sufficiently precisely to consider it as known. The following table 3 defines the acceptance/rejection rules of the lots.

Table 3: Lot acceptance/rejection criteria for σ-method

	Inspection of a minimum value L $\bar{x} \geq L$	Inspection of a maximum value U $\bar{x} \leq U$	Inspection of a range of values $L \leq \bar{x} \leq U$
Lot is accepted	$\bar{x} \geq L + K\sigma$	$\bar{x} \leq U - K\sigma$	$L + K\sigma \leq \bar{x} \leq U - K\sigma$
Lot is refused	$\bar{x} < L + K\sigma$	$\bar{x} > U - K\sigma$	$\bar{x} < L + K\sigma$, or $\bar{x} > U - K\sigma$

EXAMPLE : inspection of the maximum sodium content U of a lot of dietary cheese low in sodium for which the maximum sodium content is set by the Codex standard 53-1981 at 120 milligrams per 100 grams of commodity.

Inspected value U = 120 milligrams of sodium per 100 grams of dietary cheese

Data of the chosen sampling plan, from the Standard ISO 3951 (see Table 19):

- n = 5, number of items in the sample;
- K = 1,39, acceptance constant;
- AQL = 2,5 %.

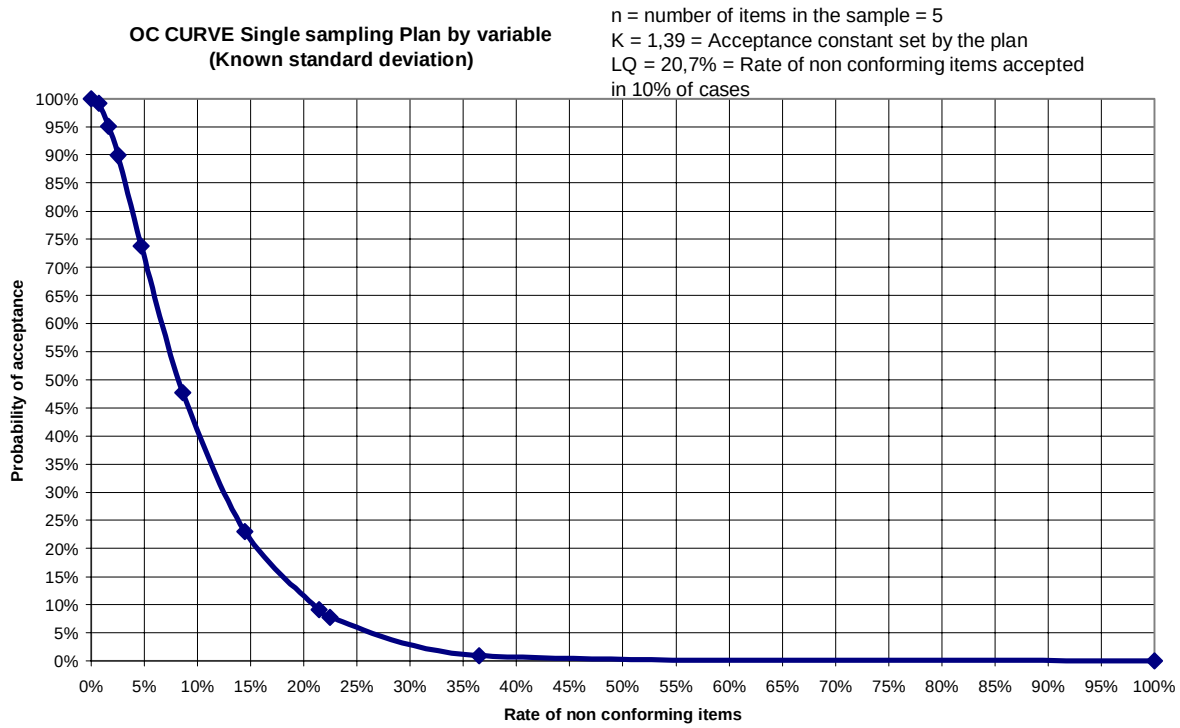
- σ = 3,5 mg, the known standard deviation according to experimental data on an extended period of production, made available to the inspectors by the professionals.

Results of measurements:

- x_1 denotes the sodium content measured in the first item, = 118 mg ;
- x_2 denotes the sodium content measured in the second item, = 123 mg ;
- x_3 denotes the sodium content measured in the third item, = 117 mg ;

- x_4 denotes the sodium content measured in the fourth item, = 121 mg ;
- x_5 denotes the sodium content measured in the fifth item, = 111 mg ;
- $\bar{x}$ denotes the mean of the sodium contents obtained on the sample of five items $\bar{x} = \frac{x_1 + x_2 + x_3 + x_4 + x_5}{5} = 118$ mg
- Conclusion: knowing that U - Kσ = 120 – (1,39 x 3,5) = 115,1 mg, then $\bar{x} > U - K\sigma$ and the lot is rejected.
- The operating characteristic curve of the plan by variables is given in the figure 2.

Figure 2: OC curve, variable sampling plan



2.5.1.2.3 The standard deviation σ of the distribution is unknown (s-method)

When the standard deviation σ of the distribution of values is unknown (for example in the case of inspections made by official inspection departments which, owing to the insufficient number of inspections they make, do not know the standard-deviation sufficiently precisely to consider it as known), the method is called the s-method, since the standard deviation σ is estimated by $s = \sqrt{\sum_{i=1}^{i=n} \frac{\left(x_i - \bar{x}\right)^2}{n-1}}$, called the standard deviation estimator (see 2.2.20).

In this case, the distribution of means calculated on the sample follows a Student distribution with n-1 degrees of freedom. The following table 4 defines the acceptance/rejection rules of the lots.

Table 4: Lot acceptance/rejection criteria for s-method

	Inspection of a minimum value L $\bar{x} \geq L$	Inspection of a maximum value U $\bar{x} \leq U$	Inspection of a range of values between L and U $L \leq \bar{x} \leq U$
Lot is accepted	$\bar{x} \geq L + Ks$	$\bar{x} \leq U - Ks$	$L + Ks \leq \bar{x} \leq U - Ks$
Lot is refused	$\bar{x} < L + Ks$	$\bar{x} > U - Ks$	$\bar{x} < L + Ks$, or $\bar{x} > U - Ks$

EXAMPLE : inspection of the maximum sodium content U of a lot of dietary cheese low in sodium for which the maximum sodium content is set by the Codex standard 53-1981 at 120 milligrams per 100 grams of commodity

Inspected value U = 120 milligrams of sodium per 100 grams of dietary cheese

Data of the chosen sampling plan, from the Standard ISO 3951 (see Table 16):

- n = 5, number of items in the sample;
- K = 1,24, acceptance constant;
- AQL = 2,5 %.

Results of measurements[13] :

- x_1 denotes the sodium content measured in the first item, = 118 mg ;
- x_2 denotes the sodium content measured in the second item, = 123 mg ;
- x_3 denotes the sodium content measured in the third item, = 117 mg ;
- x_4 denotes the sodium content measured in the fourth item, = 121 mg ;
- x_5 denotes the sodium content measured in the fifth item, = 111 mg ;
- $\bar{x}$ denotes the mean of the sodium contents obtained on the sample of five items
  $\bar{x} = \frac{x_1 + x_2 + x_3 + x_4 + x_5}{5} = 118$ mg
- s denotes the standard deviation estimator calculated on the sample :

$$s = \sqrt{\sum_{i=1}^{i=n} \frac{\left(x_i - \bar{x}\right)^2}{n-1}} = 4{,}6 \text{ mg}$$

- Conclusion : knowing that U - Ks = 120 – (1,24 x 4,6) = 114,3 mg, then $\bar{x} >$ U - Ks and the lot is rejected (see Table 3)

2.5.1.2.4 Comparison of σ and s methods

In most cases, the s-method is used, because the standard deviation is not known. In the cases of well-known and well-controlled processes, the σ-method can be used (see 2.5.1.2.2).

The difference between the two methods comes from the value of LQ (defective rate in the lots accepted in 10 % of cases), see examples of 2.5.1.2.2 and 2.5.1.2.3. In these examples:

σ-method : the LQ is 20,7 %, consequence of the characteristics of the plan (AQL = 2,5 %, n = 5, K = 1,39).

[13] In order to highlight the difference with the σ method, the numerical values are identical to whose indicated in the case of the σ method.

s-method : the LQ is 35 %, consequence of the characteristics of the plan (AQL = 2,5 %, n = 5, K = 1,24).

The following Table 5 and Figure 3 compare the efficiency of these 2 plans and show that the σ-method is more efficient that the s-method, since for the same number of items in the sample, the σ-method provides greater discrimination between good and poor quality products, ie the OC curve decreases more steeply.

Figure 3: Compared OC curves of variable sampling plans : s-method and σ-method

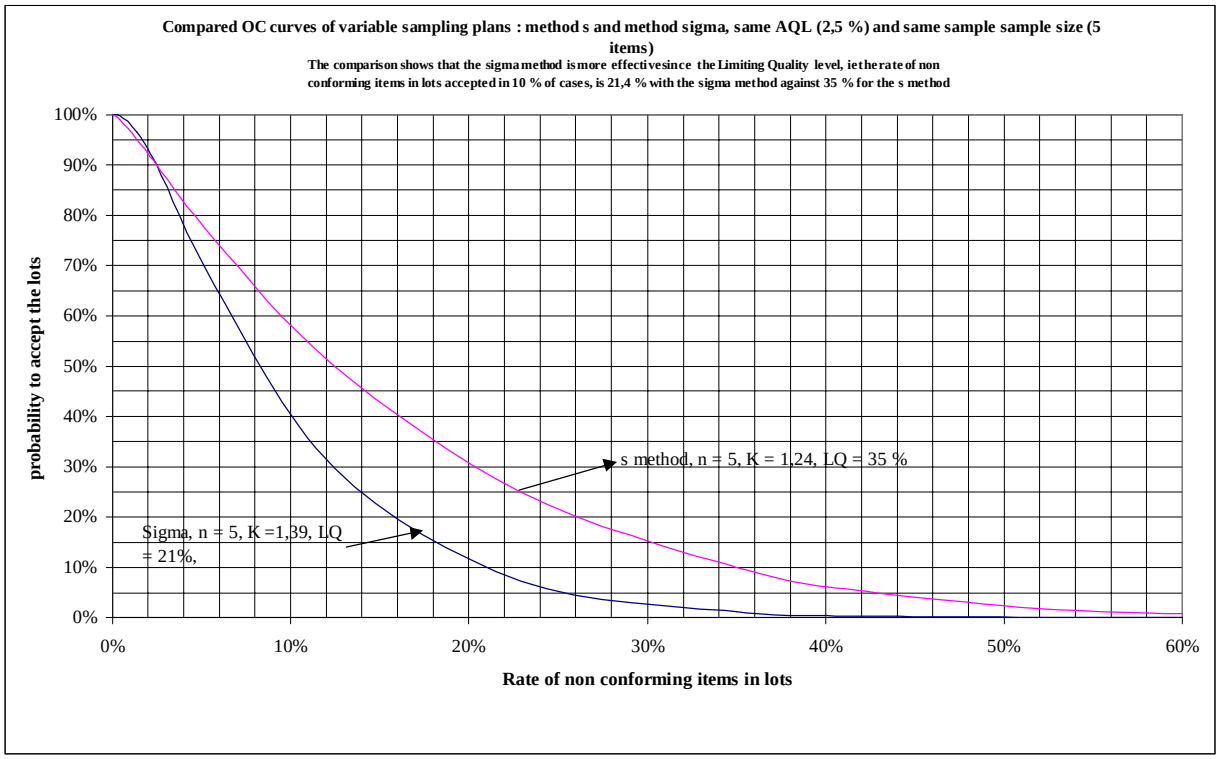


Table 5: Probability of lot acceptance by defective rates and sampling method (s-method, σ-method)

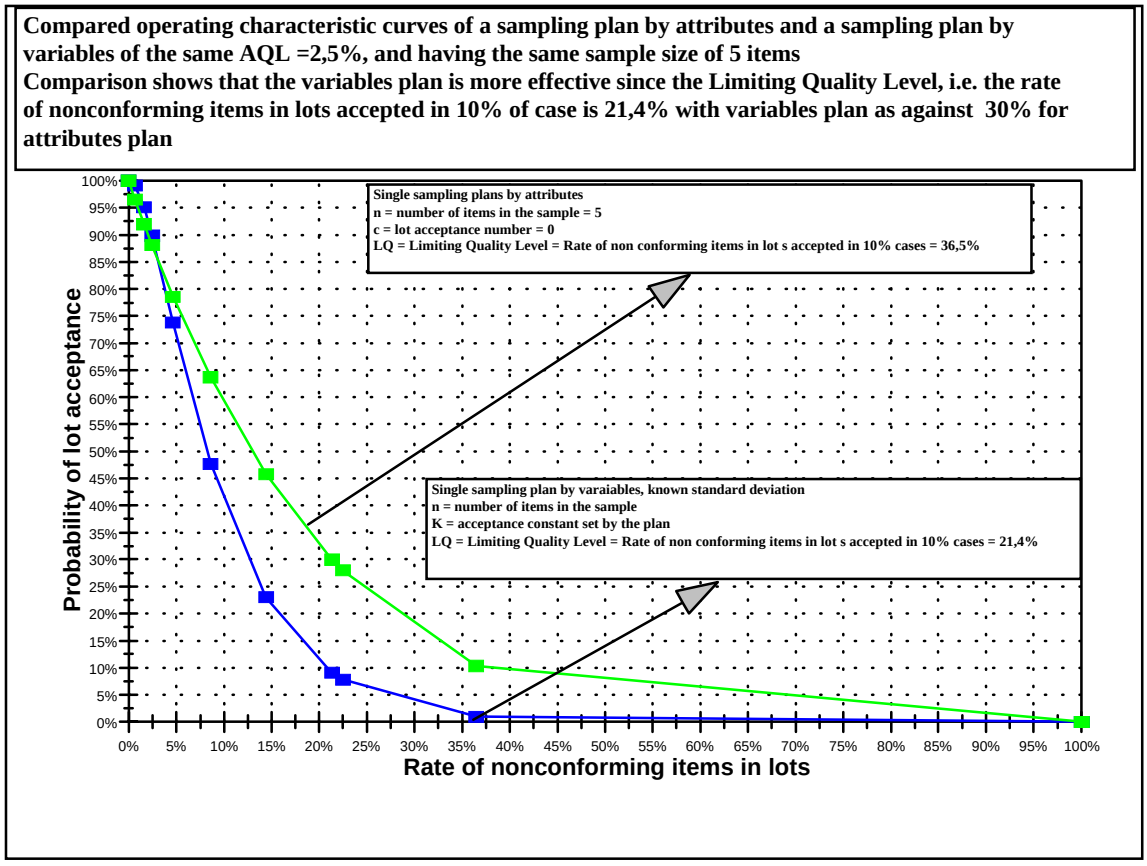
Defective rates in the lots	***Probability of lot acceptance***	
	σ -method	***s-method***
0%	*100%*	*100%*
0,4%	*99,8%*	*99%*
1,38%	*96,5%*	*95%*
2,48%	*90%*	*90%*
5,78%	*65,9%*	*75%*
12,47%	*29,7%*	*50%*
22,88%	*7,4%*	*25%*
34,98%	*1,2%*	*10%*
42,97%	*0,3%*	*5%*
58,11%	*0%*	*1%*
100%	*0%*	*0%*

2.5.1.3 Compared effectiveness of an inspection for a given defective rate by attributes and by variables

When the controlled characteristic is quantitative and normally distributed (example : control of sodium content in a dietary cheese), it is possible to use either an attribute or a variable sampling plan. Since the efficacy of an attribute sampling plan is lower (see below), it is preferable in this case to choose a variable sampling plan (see 2.5.1.4).

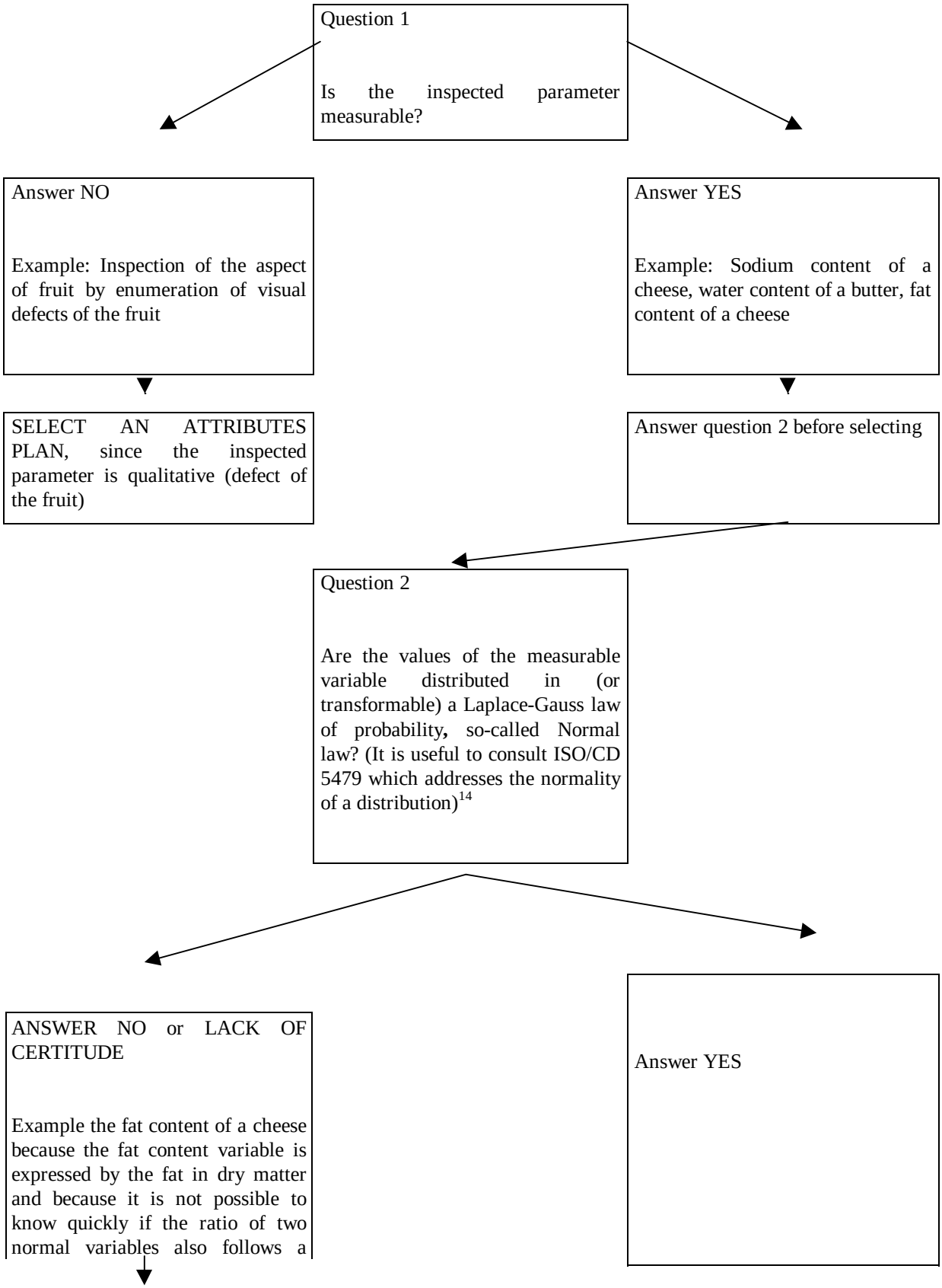
The following Figure 4 which compares the efficacy of a variable plan (σ-method) and an attribute plan, of the same AQL 2,5% and having a sample size of five items, shows that the variable plan is more effective than the attribute plan since the limiting quality of lots accepted in 10% of cases is lower with variables plans (21,4 %) than with attributes plans (36,9 %).

Figure 4: **Compared OC curves of a variable and an attribute sampling plans**



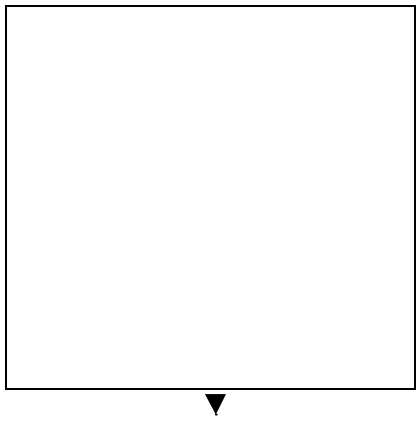
2.5.1.4 Decision tree for the selection of an attributes or a variables sampling plan

The selection of an attribute or a variable sampling plan should be made according to the following decision tree:



[14] A transformation to convert the distribution of a variable to normality should not be used, unless there is agreed documentary evidence to justify it.

normal law.



SELECT AN ATTRIBUTES PLAN, because attributes plans do not require any condition relative to the law of distribution of the values of the measurable variable

SELECT A VARIABLES PLAN because, for the same efficiency, variables plans require fewer number of items to be taken and analysed than attributes plans

2.5.1.5 Comparative advantages and disadvantages of attribute plans and variable plans

When it is possible to implement either an attributes plan or a variables plan, for example for the inspection of the sodium content of a dietary cheese, the selection must be made after having consulted in particular the following Table 6 on the comparative advantages and disadvantages of the plans[15].

Table 6: Comparison of attribute and variable sampling plans

	ADVANTAGES	DISADVANTAGES
ATTRIBUTES PLANS	No condition on the mathematical law of distribution of the variable inspected Greater simplicity of processing the results on the sample	Less effective than variables plans for a same sample size of n increments (the LQ is higher); more costly than variables plans because the collected sample requires more increments than those required, for the same efficacy, by a variables plan
VARIABLES PLANS	More effective than attributes plans for the same sample size of n increments (the LQ is lower); for the same AQL they are less expensive than attributes plans because the sample collected requires fewer increments than those required, for a same efficacy, by attributes plans	They cannot be used in all cases because to validate the calculation formulas the mathematical law of distribution of the inspected variable must necessarily follow or approximately follow a normal law

The sample sizes required when inspecting by attributes and variables are compared in the following table 7:

[15] When the inspection of two specifications, for example the fat content and the sodium content of a dietary cheese, necessitates the implementation of a plan by attributes (for the fat content) and by variables (for the sodium content), it is recommended, only for reasons of practicality of inspection, to choose a plan by attributes for the two specifications.

Table 7: Comparison of sample sizes with attribute and variable sampling plans (normal inspection level)

Sample size code letter[a]	Sample sizes	
	Inspection by attributes	Inspection by variables
C	5	4
F	20	10
H	50	20
K	125	50
N	500	150
a) From Table 1 in ISO TR 8550, the code letter gives the combinations of lot size and of "inspection levels" (section 2.2.12)		

2.5.1.6 Recommended situation for attribute sampling plans

Attributes plans are more robust than variables methods (not subject to assumptions of distributional shape) and are simpler to operate. Sampling by attributes is recommended when evaluating isolated lots. If necessary, *measurements (variables) may be converted to attributes*, in order to facilitate attribute sampling.

2.5.1.7 Recommended situation for variable plans

The variables method requires a smaller sample size than the attributes method to attain a given degree of protection against incorrect decisions - an important consideration when the sampling is destructive. However, *since each quality characteristic has to be considered separately, the variables method becomes less suitable as the number of measurements to be made on a single item increases.*

2.5.2 Zero Acceptance Number Sampling Plans

(see the Standard ISO/DIS 14 560)

This standard addresses the need for sampling plans, *based upon a zero acceptance number*, which address quality (non-conformance) levels in the parts per million (ppm or mg/kg) range within *isolated lots*. The standard does not address minor nonconformities.

Zero acceptance sampling plans in ISO/DIS 14 560 are applicable, but not limited, to inspection of (a) end items and (b) components and raw material. The selection of the appropriate plan depends upon the amount of consumer protection desired for a selected PPM level of desired product quality, and the size of the lot.

2.5.3 Sampling plans for inspection of critical nonconformities

C*ritical nonconformities* render the items hazardous, or potentially hazardous, and can result in illness or death.

2.5.3.1 Procedure of the Standard ISO 2859-0

The following procedure may be used to establish the appropriate sample size (see ISO 2859-0):

- a simple formula is used which relates :

(a) the maximum number d of critical nonconformities/nonconforming items admitted in the lot ;

(b) N the lot size;

(c) n the sample size;

(d) the risk β one is prepared to take of failing to find a nonconformity/nonconforming item, ie the probability of non detecting at least one critical nonconformity (it is usual to choose β less than or equal to 0,1 %);

(e) the probability p of maximum nonconforming items admitted in the inspected lot (p is usually taken less than or equal to 0,2 %)
p = d/N, d = Np rounded down to the nearest integer;

- the sample size n is obtained from the following equation (by rounding-up to the nearest integer):

$$n = (N - d/2)\,(1 - \beta^{1/(d+1)})$$

- the lot is accepted if no critical nonconformities are found in the sample.

EXAMPLE : Detection of defective sealed cans

Determination of sample size for the inspection of critical non confirming items (defective sealed cans) in a lot of N = 3454 cans where :

- p, the maximum percentage of nonconforming critical items, is 0,2%
- the maximum accepted risk β of accepting of non detecting a nonconforming item is 0,1%
- c, the acceptance criterion of the lot, is 0 (no nonconforming item in the sample)
- Re, the rejection criterion of the lot; is 1 (at least 1 nonconforming item in the sample).

Calculation of d : d = Np = 3454 x 0,002 = 6,908, rounded down to the nearest integer = 6

Calculation of n: $n = (N - d/2)\,(1 - \beta^{1/(d+1)}) = 2165$.

This very high value shows the great practical difficulty in using a procedure that involves destructive testing when p and β are small. The cost of such control will be high. However, it illustrates the value of applying simple non destructive, yet informative tests to every item in a lot, for example, observing whether the ends of cans are depressed, indicating a presence of an effective hermetic seal.

2.6 COST OF SAMPLING

The attention of users is drawn upon the relation between the efficiency and the size of the sample. For a given Acceptable Quality Level (AQL), the smaller the sample size, the smaller the cost of sampling, but the worse the efficiency, that is the risk to wrongly accepting a lot increases and worsens the damage in trade (in particular large financial losses .for the producer if a lot is discovered as non-compliant).

As an example, for the attributes sampling plans proposed in 4.2.2.3 (Table 13, AQL = 6,5 %) the consumers' risk (P_{10}) increases from 40,6 % (n = 8) to 68,4 % (n = 2).

The attention of users is also drawn upon the relation between the efficiency and the AQL. For a given sample size, the lower the AQL, the better the efficiency.

As an example, for a sample of 20 items, between the attribute sampling plans proposed in clause 4.2.2.1 (Table 11, AQL = 0,65 %) and in clause 4.2.2.3 (Table 13, AQL = 6,5 %), the consumers' risk (P_{10}) increases from 10,9 % to 30,4 %.

Thus for a given sample size, fixed by requirements due to the cost of analysis, the improvement of the efficiency of sampling plans requires the choice of plans corresponding to low AQL values, depending on the products.

Another possible solution for reducing the costs of sampling is to use sequential or multiple sampling plans which allows, with reduced sample size, the elimination of the lots of very low quality. These plans are out of the scope of these guidelines (see relevant ISO Standards).

SECTION 3: THE SELECTION OF SAMPLING PLANS FOR SINGLE OR ISOLATED LOTS MOVING IN INTERNATIONAL TRADE

This section presents the rationale for selecting sampling plans by attributes for single or isolated lots moving in international trade. It lays down rules for:

- inspection by attributes indexed by the limiting quality (LQ) level (section 3.1)
- inspection by two or three class attributes for microbiological assessments (section 3.2)

3.1 SAMPLING PROCEDURES FOR INSPECTION BY ATTRIBUTES: SAMPLING PLANS INDEXED BY LIMITING QUALITY (LQ) FOR ISOLATED LOT INSPECTION

(see ISO 2859/2-1985 (E))

Preliminary note[16] : Given the requirements due to probabilities linked to sampling by attributes, the plans of this section enable a rational choice between the existing plans referring to AQL, as defined in Section 4.2. In order to ensure their compatibility, similar rules for acceptance/rejection, as well as categories of lot size have been chosen for this section and for section 4.2.

This ISO Standard provides sampling plans for application to single lots (procedure A, 3.1.1) or to lots isolated from a series (procedure B, 3.1.2) *where the 'switching rules' (see Section 2.2.16) are precluded*. Both procedures use the limiting quality (LQ; Section 2.2.5) as an indicator of the actual percentage nonconforming in the lots submitted. The associated Consumer's Risk (the probability of accepting a lot with the limiting quality level) is usually less than 10 per cent, but always below 13 per cent.

Procedure A is used when *both the producer and consumer wish to regard the lot in isolation*; and *it is also used as the default procedure* (i.e. it is used unless there is a specific instruction to use procedure B). Procedure A includes plans *with acceptance number zero*, and with sample sizes based upon the hypergeometric distribution of sampling results. **Procedure B** is used when *the producer regards the lot as one of a continuing series, but the consumer considers the lot in isolation*. This approach allows the producer to maintain consistent production procedures for a variety of consumers whilst any individual consumer is concerned with only one particular lot. Procedure B excludes plans with zero acceptance numbers, replacing them with one hundred percent evaluation.

Procedures A and B may be compared as follows:

Procedure A (default procedure)	Procedure B
Producer & consumer regard lot in isolation	Producer regards lot as one of continuing series Consumer regards lot in isolation
Identified by lot size and LQ	Identified by lot size, LQ & inspection level
Includes plans with an acceptance number of zero	Plans with an acceptance number of zero not included
Double & multiple plans can be used as alternatives to zero acceptance number plans	Double & multiple plans can be used as alternatives to single sampling plans

3.1.1 Procedure A: Producer and consumer regard lot in isolation

The application of procedure A may be illustrated as follows:

Summary of sampling plan

Set LQ

⬇

Select sample size (n) & acceptance number (c) (Table A in ISO 2859/2-1985 (E)) and collect sample

⬇

Inspect each item in the sample

⬇

Accept the lot if: number of nonconforming items ≤ c

[16] According to 7.1 of Standard ISO 2859-2.

3.1.2 Procedure B: Producer regards lot as one of a continuing series: Consumer regards lot in isolation

The application of procedure B may be summarised as follows:

Summary of sampling plans

Set LQ

⬇

Select inspection level
(Table I in ISO 2859-1 : 1989 (E) and Table B6 in ISO 2859/2-1985(E))

⬇

Select sample size, n & acceptance number, c (Tables B1-B10, ISO 2859/2-1985(E))
and collect sample

⬇

Inspect each item in the sample

⬇

Accept the lot if: number of nonconforming items ≤ c

3.2 TWO AND THREE CLASS ATTRIBUTES PLANS FOR MICROBIOLOGICAL ASSESSMENTS (SEE REFERENCE 6.1)

3.2.1 Two-class Attributes Plans

Two-class attributes plans provide a simple means of inspection *where the sampling plan is defined by two values, n and c*. The value of n defines the sample size in terms of the number of items; and the value c denotes the maximum number of nonconforming items permitted in the sample. When undertaking a microbiological assessment, a maximum concentration of micro-organisms permitted in any item is denoted by m; any item contaminated at a concentration greater than m is considered to be nonconforming.

For a given value of c, the stringency (probability of rejection) of the plan will increase as n increases. Similarly, for a given value of n, the stringency will increase as c decreases. The equation of the OC of such plans is the following :

$$P_A = P\,[x \leq c] = \sum_{i=0}^{i=c} C_n^i p^i (1-p)^{n-i}$$

Where :

P_A = Probability to accept the lot

p = Defective rate in the lot, ie lots for whose the concentration of micro-organisms is greater than m

i and x are whole discrete variables, varying between 0 and c

$$C_n^i = \frac{n!}{i!(n-i)!}$$

The application of a two-class attributes plan can be summarized as follows :

Set the value of m, n and c

⬇

Collect the sample with n items

⬇

Inspect each item in the sample

⬇

Accept the lot if: number of defective items ≤ c

EXAMPLE : Inspection of the presence of *Salmonella* in fresh vegetables

- Description of an ICMSF plan :

n = 5 = number of items of 25 g in the sample

m = maximum content admitted in *Salmonella* per item = 0 CFU in 25 g

c = 0 = maximum number of items of the sample where the concentration x in *Salmonella* is higher than m (ie *Salmonella* is detected).

The lot is accepted if no item in the sample shows a presence of *Salmonella*. The lot is rejected in the opposite case.

- Result of the inspection :

The results of the detections in the sample are the following:

x_1 = Salmonella detected

$x_2 = 0$

$x_3 = 0$

$x_4 = 0$

$x_5 = 0$

There is one item where *Salmonella* was detected (ie whose concentration in *Salmonella* is greater than m), the lot is therefore rejected.

3.2.2 Three-class Attributes Plans[17]

Three class attributes plans are defined by the values n, c, m and M (see below); and are applied to situations where the *quality of the product can be divided into three attribute classes* depending upon the concentration of micro-organisms within the sample:

- unacceptable quality, with a concentration of micro-organisms above the value, M (which must not be exceeded by any items in the sample).
- good quality, where the concentration must not exceed the value, m.
- marginally acceptable quality. Marginal items have a concentration which exceeds m, but which is less than M (such concentrations are undesirable but some can be accepted, the maximum number acceptable being denoted by c).

The value m is the concentration of the micro-organism which is acceptable and attainable in the food under inspection, as reflected by Good Commercial Practice (GCP). For 3-class plans, m will be assigned a non-zero value.

The value M is a hazardous or unacceptable level of contamination caused by poor hygienic practice, including improper storage. There are several approaches to choosing the value of M:

---

[17] For inhomogeneous lots (especially the ones where the distribution of the characteristic shows several peaks), a a stratified sampling plan should be performed.

(i) as a 'utility' (spoilage or shelf-life) index, relating levels of contamination to detectable spoilage (odour, flavour) or to an unacceptably short shelf-life;

(ii) as a general hygiene indicator, relating levels of the indicator contaminant to a clearly unacceptable condition of hygiene;

(iii) as a health hazard, relating contamination levels to illness. A variety of data may be used for this purpose including, for example, epidemiological, experimental animal feeding and human feeding data.

The values m and M may be independent of each other.

The choice of values for n and c varies with the desired stringency (probability of rejection). For stringent 'cases', n is high and c is low; for lenient 'cases' n is low and c is high. The choice of n is usually a compromise between what is an ideal probability of assurance of consumer safety and the work load the laboratory can handle.

If the concentration of micro-organisms in any item of the sample is greater than M, the lot is directly rejected.

The equation of the OC curve of such plans is the following :

$$P_a = \sum_{i=0}^{i=c} C_n^i \left(\frac{P_m}{100}\right)^i \left(\frac{100 - P_d - P_m}{100}\right)^{n-i}$$

where :

P_a is the probability of acceptance of a lot containing :

- a given percentage of defective items (P_d) (a defective item having a concentration in micro-organisms greater than M), i.e. lots for whose the concentration in micro-organisms is greater than M), and
- a given percentage of marginally acceptable items (P_m) (a marginally acceptable item having a concentration in micro-organisms between m and M) ;

n is the number of items in the sample

c is the maximum number allowed of marginal items.

The application of a three-class attributes sampling plan may be summarized as follows :

Set the values of m, M, n ,c

⬇

Collect the sample with n items

⬇

Inspect each item in the sample

⬇

Accept the lot if: number of marginally defective items (i.e. a concentration of micro-organisms between m and M) ≤ c

Immediately reject the lot if the concentration of micro-organisms in any item > M and/or the number of marginally defective items > c.

EXAMPLE : Inspection of the concentration of mesophilic aerobic micro-organisms in fresh vegetable

- Description of an ICSMF plan :

n = 5 = the number of items in the sample

m = 10^6 CFU/g

M = 5 10^7 CFU/g

c = 2 = the maximum number allowed of items in the sample whose concentration in mesophilic aerobic micro-organisms lies between m and M

The lot is accepted if no item shows a concentration greater than M and if the maximum number of items in the sample whose concentration lies between m and M, is at most egal to c.

- Result of the inspection

The measures of concentration in the sample are the following :

$x_1 = 2.\ 10^7$
$x_2 = 2.10^6$
$x_3 = 2.\ 10^7$
$x_4 = 2.10^6$
$x_5 = 2.10^6$

There are 5 items of the sample whose concentration in mesophilic aerobic micro-organisms lies between m and M, this figure is greater than c and the lot is rejected.

3.2.3 The Application of Two and Three-class Attributes Plans

Two and three-class attributes plans are ideally suited for regulatory, port-of-entry, and other consumer-oriented situations where little information is available concerning the microbiological history of the lot. The plans are independent of lot size if the lot is large in comparison to sample size. The relationship between sample size and lot size only becomes significant when the sample size approaches one tenth of the lot size, a situation rarely occurring in the bacteriological inspection of foods.

When choosing a plan one must consider: (i) the type and seriousness of hazards implied by the micro-organisms; and (ii) the conditions under which the food is expected to be handled and consumed after sampling. Table 8 (after Table 10 of the ICMSF publication) classifies 15 different 'cases' of sampling plans taking these factors into consideration, the stringency of the plans increasing with the type and degree of hazard. Case 1 requires the most lenient plan whereas Case 15 represents the most stringent requirement. In Table 8, a sampling plan is recommended for each of the 15 'cases'.

Table 8: Classification of sampling plans according to nature of concern and hazard

Nature of concern	Decreased hazard	Unchanged hazard	Increased hazard
No direct health hazard (spoilage and shelf-life)	n = 5, c= 3	n = 5, c = 2	n = 5, c = 1
Low indirect health hazard (indicator organisms)	n = 5, c= 3	n = 5, c = 2	n = 5, c = 1
Moderate direct health hazard (limited spread)	n = 5, c= 2	n = 5, c = 1	n = 10, c = 1
Moderate direct health hazard of potentially extensive spread in food	n = 5, c= 0	n = 10, c = 0	n = 20, c = 0
Severe direct health hazard	n = 15, c= 0	n = 30, c = 0	n = 60, c = 0

EXAMPLES :

(i) A sampling plan is required for the inspection of fresh or frozen fish for the bacterium *Escherichia coli*. The contamination of fish with *E. coli* is considered (1) to be a low indirect health hazard which is likely to be reduced during the handling of the fish. Normally the fish will be cooked before consumption. Consequently, the contamination of fish with E. coli may be classified as Case 4 in Table 10 and the recommended sampling plan is a 3-class attributes plan, where n = 5 and c = 3. (The values of m and M will also be specified.)

(ii) The contamination of cooked crabmeat with *Staphylococcus aureus* is considered (1) to be a moderate direct health hazard of limited spread which is likely to increase with handling (Case

9). Consequently, the appropriate sampling plan for the inspection of *S. aureus* in cooked crabmeat is a 3-class plan where n = 10 and c = 1. (The values of m and M will also be specified.)

(iii) The contamination of frozen, ready-to-eat, bakery products (with low-acid or high water activity fillings or toppings) with *Salmonella* is considered to be a moderate direct health hazard of potentially extensive spread in food which is likely to increase with handling (Case 12). In this example, the appropriate plan is a 2-class plan where n = 20 and c = 0.

3.3 SINGLE SAMPLING PLANS FOR AVERAGE CONTROL (STANDARD DEVIATION UNKNOWN)

Such a control is performed by using a test which aims at ensuring that, on average, the content of the controlled characteristic is at least equal to either the quantity given of the label of the product, or the quantity fixed by the regulation or a code of practice (e.g. net weight, net volume,…).

Description of the test

n is the sample size, in number of items, used for the test

$\bar{x} = \frac{\sum_{i=1}^{n} x_i}{n}$ is the sample mean of the n items in the sample

$s = \sqrt{\frac{\sum_{i=1}^{n} \left(x_i - \bar{x}\right)^2}{n-1}}$ is the standard deviation of the values of the items in the sample.

α is the significance level of the test, that is the probability of wrongly concluding that the mean content of the controlled chacteristic is less than the stated value when it is indeed greater than or equal to that value.

t_α is the value of the Student's t-distribution, on n-1 degrees of freedom, corresponding to the significance level α[18].

M is the stated value for the mean of the lot.

Decision Rules

The lot is accepted if:

$\bar{x} \geq M - \frac{t_\alpha \times s}{\sqrt{n}}$

and rejected otherwise.

[18] α is generally taken at 5%, or 0,5%.

The following Table provides t-values of the Student's distribution for some selected sample sizes and for α of 5 % and 0,5 %.

Number of Samples	t-value (α = 5%)	t-value (α = 0,5%)
5	2,13	4,60
10	1,83	3,25
15	1,76	2,98
20	1,73	2,86
25	1,71	2,80
30	1,70	2,76
35	1,69	2,73
40	1,68	2,71
45	1,68	2,69
50	1,68	2,68

SECTION 4. THE SELECTION OF SAMPLING PLANS FOR A CONTINUOUS SERIES OF LOTS FROM A SINGLE SOURCE

4.1 PRESENTATION OF SECTION 4

Normally, the sampling plans described in Sections 4.2 and 4.3 should only be applied to a continuous series of lots from a single source. However, the plans described below (including the switching rules) may be utilised when data have been collected describing the quality of isolated lots, from a single source, over a prolonged period of time.

This section addresses the selection of single sampling plans for inspection of percent nonconforming, for a continuing series of lots coming from a single source.

It recommends single sampling plans by attributes (section 4.2) and by variables (section 4.3)[19] with their characteristics:

- Number of items in the sample,
- Acceptable Quality Level (AQL),
- for attributes plans: acceptance number c, i.e. the maximum number of nonconforming items in the sample,
- for variables plans, the acceptance constant K to be included in the lot acceptance formula,
- operating characteristic curves.

To make the document readily readable, and to achieve minimum difficulty in implementing the plans and minimum inspection cost, these plans are limited to the following characteristics:

- AQL 0.65%,. 2.5%, , 6.5%
- n, number of items in the sample, included between 2 and 50
- P_{10} = Rate of non-conforming items in lots accepted in 10% of cases = LQ
- P_{50} = Rate of non-conforming items in lots accepted in 50% of cases
- P_{95} = Rate of non-conforming items in lots accepted in 95% of cases

[19] The plans of Section 4.3.2 may also be used for isolated lots.

Codex Committees and, where applicable, governments, will select from these plans on the basis of the quality aim they set themselves. This quality level is stated by the Acceptable Quality Level.

The lowest level of acceptable quality or LQ derives from the characteristics of the choice of n and of AQL.

Each single sampling plan recommended in section 4 is accompanied by a table giving the plan characteristics (AQL, n = sample size, : c = acceptance number of the lot, in the case of plans by attributes, K = acceptance constant, in the case of plans by variables) and the probability of lot acceptance as a function of the rate of nonconforming items in these lots, particularly the LQ or rate of nonconforming items in lots accepted in 10% of cases. All the plans recommended according to the AQL and the size n of the sample, are also grouped per AQL in a graph like the Figure 5, of the Operating Characteristic (OC) curve, which relates the rate of nonconforming items in an inspected lot and the probability of lot acceptance.

The following example illustrates this principle of presentation of recommended plans with tables (Table 9) and graphs (Figure 5) of OC curves for simple sampling plans by attributes, of AQL = 6,5 %, n= 2, c = 0 and n = 50, c = 7.

Table 9: Probability of lot acceptance, attribute sampling plan, AQL = 6,5 %

Defective rates in the lots	**Probability of lot acceptance**					
	n = 2, c = 0 P_{95} = 2,53% P_{50} =29,3% P_{10} =68,4%	**n = 8, c= 1** P_{95} = 2,64% P_{50} =20% P_{10} = 40,6%	**n = 13, c= 2** P_{95} = 6,63% P_{50} =20% P_{10} = 36%	**n = 20, c= 3** P_{95} = 7,13% P_{50}=18,1% P_{10}= 30,4%	**n = 32, c= 5** P_{95} = 8,5% P_{50} =17,5% P_{10} = 27,1%	**n = 50, c= 7** P_{95} =8,2% P_{50} =15,2% P_{10} = 22,4%
0%	100%	100%	100%	100%	100%	100%
5 %	90,3%	94,3%	97,5%	98,4%	99 %	99,7%
6,5%	87,4%	90,9%	95,2%	96,3%	98,4%	98,5%
10 %	81%	81,3%	86,6%	86,7%	90,6%	87,8%
20%	64%	50%	50%	41,1%	36%	19%
30 %	49%	25,5%	20,2%	10,7%	5,1%	0,7%
40%	36%	10,6%	5,8%	1,6%	0,3%	0%
50%	25%	3,5%	1,1%	0,1%	0%	0%
60 %	16%	0,9%	0,1%	0%	0%	0%
80%	4,0%	0%	0%	0%	0%	0%
90%	1%	0%	0%	0%	0%	0%
100%	0%	0%	0%	0%	0%	0%

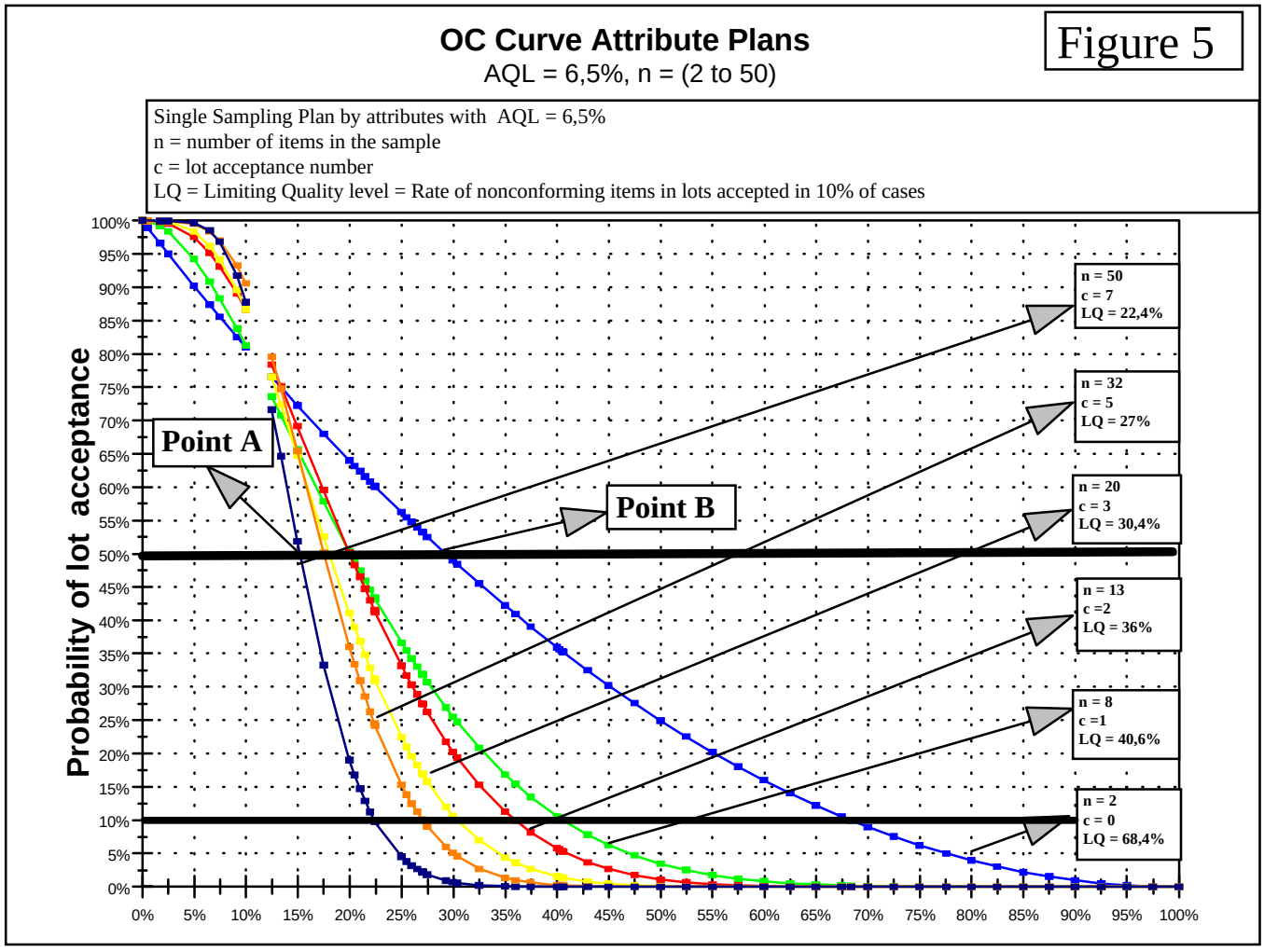
Figure 5 gathers the OC curves of these plans by attributes, fixed by the Standard ISO 2859-1.

The curve of Figure 5, which contains the point A, corresponds to a lot inspected with a 50-item sample. The lot is accepted at inspection if there are less than 7 defective items in the sample. The abscissa of the point A (15 %) corresponds to a lot containing 15 % of defective items, its ordinate (50 %) corresponds to the probability to accept these lots containing 15 % of defective items.

The curve of Figure 5, which contains the point B, corresponds to a lot inspected with a 2-item sample. The lot is accepted at inspection if there are less than 0 defective items in the sample. The abscissa of the point B (30 %) corresponds to a lot containing 30 % of defective items, its ordinate (50 %) corresponds to the probability to accept these lots containing 30 % of defective items.

The graph shows that, for a constant AQL, the higher the sample size, the smaller the risk to the consumer of accepting lots with high defective rates.

Figure 5: OC curve, attribute sampling plan, AQL = 6,5 %



Examples of sampling plans covering frequent inspection situations using AQL = 0,65 % or 2,5 % or 6,5 % are presented in 4.2.2.1 to 4.2.2.3.

4.2 SINGLE SAMPLING PLANS RECOMMENDED FOR INSPECTION OF DEFECTIVE PERCENTAGE BY ATTRIBUTES (FROM ISO 2859-1 : 1989)

4.2.1 General

The principle of such sampling plans is presented in Section 2.5.1.1.

The application of ISO 2859-1 attributes sampling plans may be summarised as follows:

Set inspection level
(normal[20], tightened, reduced)

⬇

Set the AQL

⬇

Select sample size, n of the sample and the acceptance number, c and collect the sample

⬇

Inspect each item in the sample and enumerate each nonconforming item in the sample

⬇

Accept the lot if this number of nonconforming items ≤ c

4.2.2 Recommended plans by attributes

This document recommends the following simple sampling plans, for covering frequent inspection situations. They are extracted from the Standard ISO 2859-1, and are characterised by their **AQL** (AQL of 0,65 %, 2,5 % and 6,5 % covering the most frequent cases), the **size n of items** in the sample and c the acceptance criterion which defines the maximum number of defective items allowed in the sample for accepting the lot. Each plan is accompanied by a table which gives the probability to accept the lots in function of the defective rate in these lots. For each AQL, a graph shows the OC curves of the corresponding recommended plans.

The OC curves have been built point-by-point from the following equation :

$$P_A = P\,[x \leq c] = \sum_{i=0}^{i=c} C_n^i p^i (1-p)^{n-i}$$

Where :

P_A = probability to accept the lot

p = defective rate in the lot

i and x are discrete whole variables, between 0 and c

$$C_n^i = \frac{n!}{i!(n-i)!}$$

Table 10 (from NMKL Procedure N° 12, see reference 5) describes the number of items to be sampled at different inspection levels, lot sizes and acceptance numbers at AQL of 0,65%, 2,5% and 6,5% respectively. The table is a simplification of a single attribute sampling plan from ISO 2859-1. This table considers three levels of inspection: tightened, normal and reduced (see 2.2.16).

[20] Any inspection level other than the normal control shall be justified by the users of sampling plans.

Table 10. Attribute Sampling Plan

	Inspection level			
Lot size		Reduced	Normal	Tightened
2-8	n	**2**	**2**	**3**
	c at AQL = 0,65	0	0	0
	c at AQL = 2,5	0	0	0
	c at AQL = 6,5	0	0	0
9-15	n	**2**	**3**	**5**
	c at AQL = 0,65	0	0	0
	c at AQL = 2,5	0	0	0
	c at AQL = 6,5	0	0	1
16-25	n	**2**	**5**	**8**
	c at AQL = 0,65	0	0	0
	c at AQL = 2,5	0	0	0
	c at AQL = 6,5	0	1	1
26-50	n	**2**	**8**	**13**
	c at AQL = 0,65	0	0	0
	c at AQL = 2,5	0	0	1
	c at AQL = 6,5	0	1	1
51 - 90	n	**2**	**13**	**20**
	c at AQL = 0,65	0	0	0
	c at AQL = 2,5	0	1	1
	c at AQL = 6,5	0	2	2
91 - 150	n	**3**	**20**	**32**
	c at AQL = 0,65	0	0	0
	c at AQL = 2,5	0	1	1
	c at AQL = 6,5	0	3	3
151 - 280	n	**5**	**32**	**50**
	c at AQL = 0,65	0	0	1
	c at AQL = 2,5	0	2	2
	c at AQL = 6,5	1	5	5
281 - 500	n	**8**	**50**	**80**
	c at AQL = 0,65	0	1	1
	c at AQL = 2,5	0	3	3
	c at AQL = 6,5	1	7	8
501 - 1 200	n	**13**	**80**	**125**
	c at AQL = 0,65	0	1	1
	c at AQL = 2,5	1	5	5
	c at AQL = 6,5	2	10	12
1 201 – 1 320	n	**20**	**125**	**200**
	c at AQL = 0,65	1	2	2
	c at AQL = 2,5	1	7	8
	c at AQL = 6,5	3	14	18
1 321 – 10 000	n	**32**	**200**	**315**
	c at AQL = 0,65	0	3	3
	c at AQL = 2,5	2	10	12
	c at AQL = 6,5	5	21	18

Table 10 (continued)

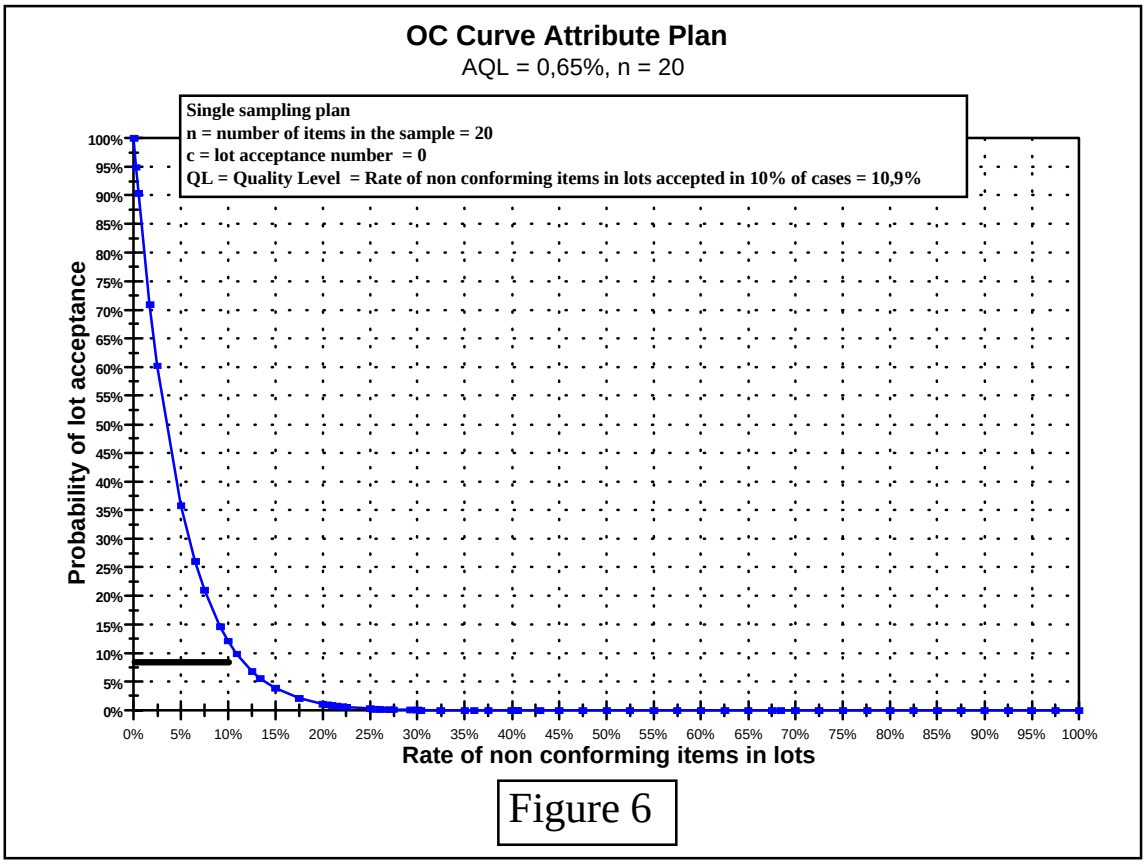
10 001 – 35 000	n	**50**	**315**	**500**
	c at AQL = 0,65	1	5	5
	c at AQL = 2,5	3	14	18
	c at AQL = 6,5	7	21	18
35 001 - 150 000	n	**80**	**500**	**800**
	c at AQL = 0,65	1	7	8
	c at AQL = 2,5	5	21	18
	c at AQL = 6,5	10	21	18
150 001 - 500 000	n	**125**	**800**	**1 250**
	c at AQL = 0,65	2	10	12
	c at AQL = 2,5	7	21	18
	c at AQL = 6,5	12	21	18
500 001 and over	n	**200**	**1 250**	**2 000**
	c at AQL = 0,65	3	14	18
	c at AQL = 2,5	10	21	18
	c at AQL = 6,5	12	21	18

4.2.2.1 Plans with AQL = 0,65 % (see Table 11 and Figure 6)

Table 11: Probability of lot acceptance, attribute sampling plans, AQL = 0,65 %

Defective rates in the lots	Probability of lot acceptance Normal inspection plan Letter-code F, AQL = 0,65%, n= 20, c =0
0%	100%
0,05%	99%
0,25%	95%
0,525%	90%
0,65%	87,8%
1,43%	75%
3,41%	50%
5%	35,8%
6,7%	25%
10%	12,2%
10,9%	10%
13,9%	5%
15%	3,9%
20%	1,2%
20,6%	1%
30%	0,1%
35%	0%
100%	0%

Figure 6: OC curve, attribute sampling plan, AQL = 0,65 %



4.2.2.2 Plans with AQL = 2,5% (see Table 12 and figure 7)

Table 12: Lot acceptance probability for AQL = 2,5 %

Defective rates in the lots	Probability of lot acceptance Normal inspection plan			
	Letter-code C, AQL = 2,5%, n= 5, c =0 P_{95} = 1,02% P_{50} =12,2% P_{10} = 36,9%	Letter-code F, AQL = 2,5%, n= 20, c =1 P_{95} = 1,8% P_{50} =8,25% P_{10} = 18,1%	Letter-code G, AQL = 2,5%, n= 32, c =2 P_{95} = 2,59% P_{50} =8,25% P_{10} = 15,8%	Letter-code H, AQL = 2,5%, n= 50, c =3 P_{95} = 2,77% P_{50} =7,29% P_{10} = 12,9%
0%	100%	100%	100%	100%
1%	95%	98,3%	99,6%	99,8%
2,5%	88,1%	91,2%	95,5%	96,4%
5%	77,4%	73,6%	78,6%	76%
10%	59%	39,2%	36,7%	25%
15%	44,4%	17,6%	12,2%	4,6%
20%	32,8%	6,9%	3,2%	0,6%
30%	16,8%	0,8%	0,1%	0%
40%	7,8%	0,1%	0%	0%
50%	3,1%	0%	0%	0%
²100%	0%	0%	0%	0%

Figure 7: OC curve, attribute sampling plan, AQL = 2,5 %

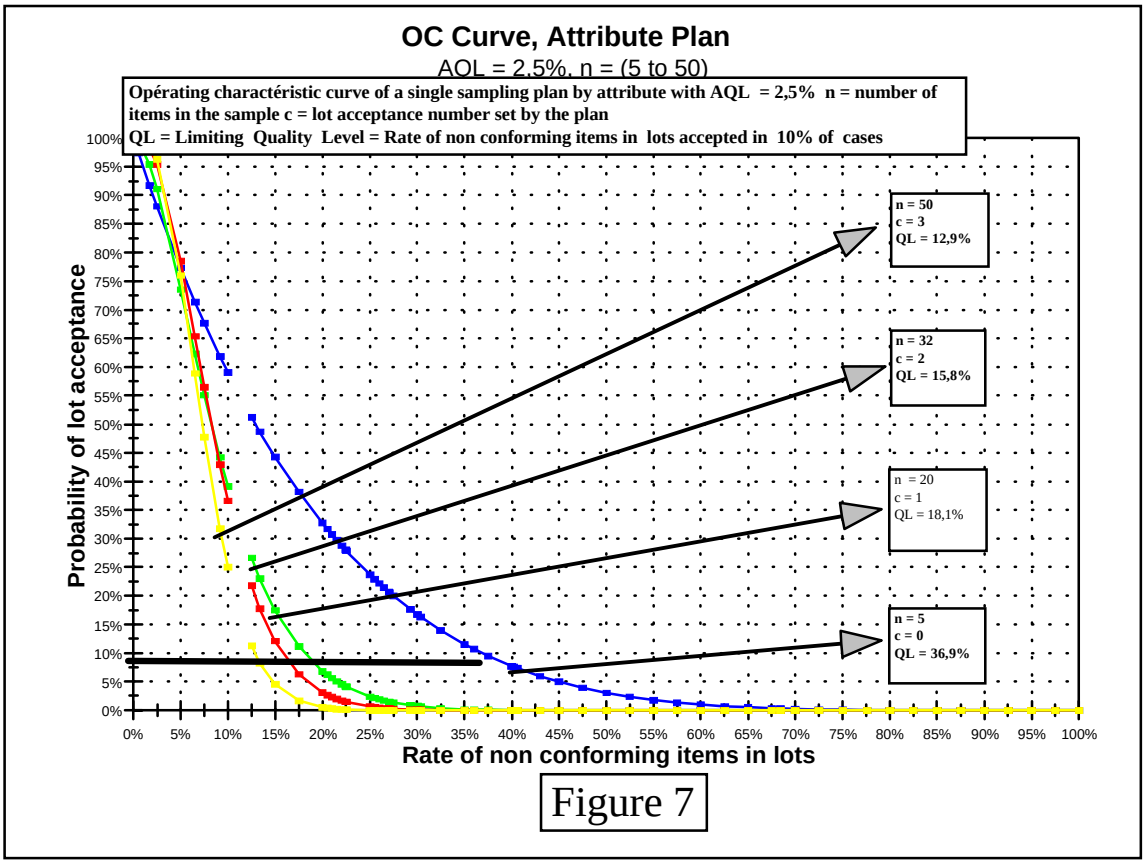


Figure 7

4.2.2.3 Plans at AQL = 6,5 % (see table 13 and figure 8)

Table 13: Probability of lot acceptance at AQL = 6,5 %

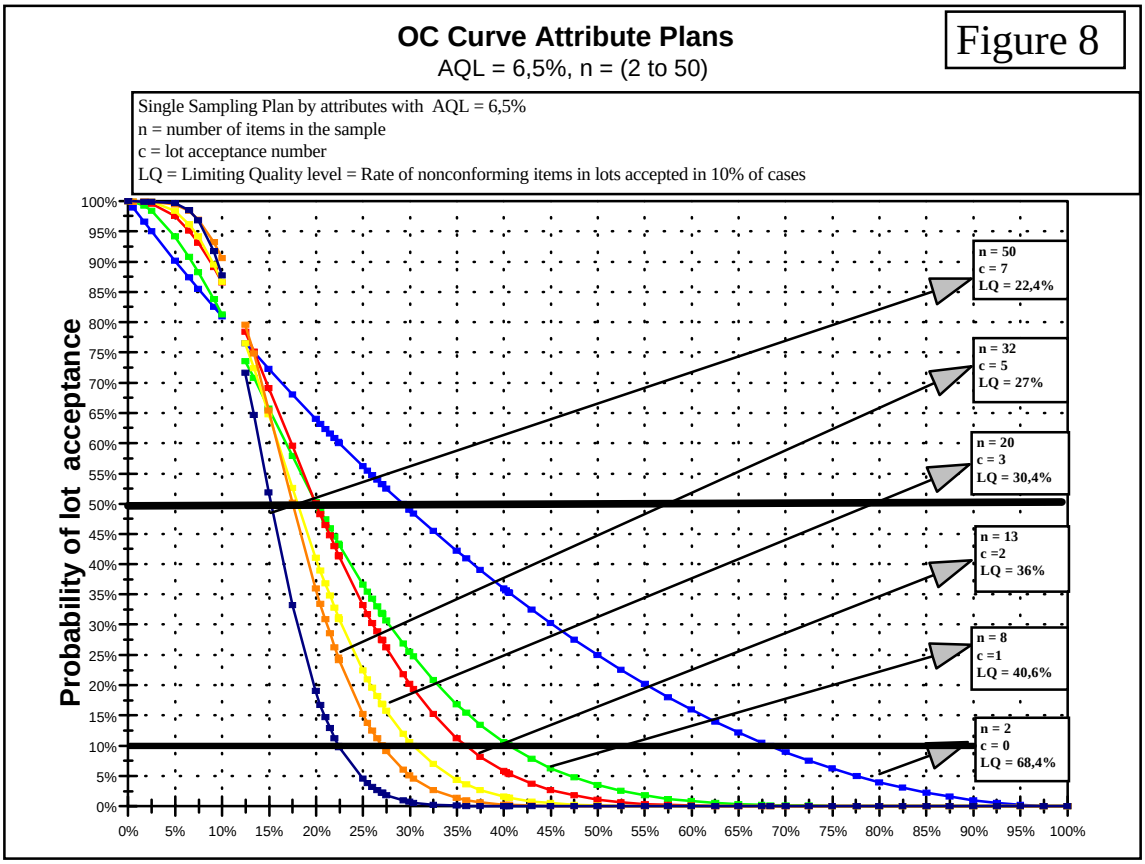
Defective rates in the lots	Probability of lot acceptance Normal inspection plan					
	Letter-code A, AQL=6,5% n= 2, c =0 P_{95} [21]= 2,53% P_{50} [22]=29,3% P_{10}[23] =68,4%	Letter-code D, AQL =6,5% n= 8, c =1 P_{95} = 2,64% P_{50} =20% P_{10} = 40,6%	Letter-code E, AQL =6,5% n= 13, c =2 P_{95} = 6,63% P_{50} =20% P_{10} = 36%	Letter-code F, AQL =6,5% n= 20, c =3 P_{95} = 7,13% P_{50}=18,1% P_{10}= 30,4%	Letter-code G, AQL =6,5% n= 32, c =5 P_{95} = 8,5% P_{50} =17,5% P_{10} = 27,1%	Letter-code H, AQL =6,5% n= 50, c =7 P_{95} =8,2% P_{50} =15,2% P_{10} = 22,4%
0%	100%	100%	100%	100%	100%	100%
5 %	90,3%	94,3%	97,5%	98,4%	99,1%	99,7%
6,5%	**87,4%**	**90,9%**	**95,2%**	**96,3%**	**98,4%**	**98,5%**
10 %	81%	81,3%	86,6%	86,7%	90,6%	87,8%
20%	64%	**50%**	**50%**	41,1%	36%	19%
30 %	49%	25,5%	20,2%	10,7%	5,1%	0,7%
40%	36%	10,6%	5,8%	1,6%	0,3%	0%
50%	25%	3,5%	1,1%	0,1%	0%	0%
60 %	16%	0,9%	0,1%	0%	0%	0%
80%	4,0%	0%	0%	0%	0%	0%
90%	1%	0%	0%	0%	0%	0%
100%	0%	0%	0%	0%	0%	0%

[21] P_{95} = Rate of non-conforming items in lots accepted in 95% of cases

[22] P_{50} = Rate of non-conforming items in lots accepted in 50% of cases

[23] P_{10} = Rate of non-conforming items in lots accepted in 10% of cases

Figure 8: OC curve, attribute sampling plan, AQL = 6,5 %



4.2.2.4 Switching Rules and Procedures (see clause 9.3; ISO 2859-1:1989(E))

Tightened Inspection

When normal inspection is being performed, tightened inspection must be introduced when two out of five, or less, consecutive lots have been non-acceptable on original inspection (ignoring resubmitted lots). Normal inspection can only be restored when five successive lots have been accepted under tightened inspection.

When operating under tightened inspection, an appropriate sampling plan is selected using the procedure described in Section 4.1, excepting that Table II-B in ISO 2859-1: 1989 (E) is used for the selection of n and Ac. In general, a tightened plan has the same sample size as the corresponding normal plan but a smaller acceptance number. However, if the normal inspection acceptance number is 1 or 0, tightening is achieved by retaining the acceptance number whilst increasing the sample size.

Reduced Inspection

When normal inspection is being performed, reduced inspection may be operated provided that each of the following conditions is satisfied:

(a) the preceding 10 lots (or more) have been subjected to normal inspection and all have been accepted on original inspection; and

(b) the total number of nonconforming units (or nonconformities) in the samples from the preceding 10 lots (or such other number as was used for condition (a), above) is equal to or less than the appropriate 'limit number' given in Table VIII in ISO 2859-1: 1989 (E); and

(c) production is at a 'steady state' (ie there has not been a break in production sufficient to invalidate the argument that the present quality is good because the record of the recent past is good, and that all factors which are likely to effect the quality of the product have remained consistent); and

(d) reduced inspection is considered desirable by the responsible authority.

In these circumstances, the inspection costs may be reduced by using reduced-inspection sampling plans which, typically, have sample sizes only two-fifths the size of the corresponding normal inspection plans. When operating under reduced inspection, an appropriate sampling plan is selected using the procedure described in Section 4.1, excepting that Table II-C in ISO 2859-1: 1989 (E)is used for the selection of n and Ac.

Normal inspection should be reverted to if a lot is not accepted on reduced inspection; or if production becomes irregular or delayed; or if other conditions occur which are likely to invalidate the steady-state condition.

Discontinuation of Inspection

Once tightened inspection has been introduced, the acceptance procedures of ISO 2859 should be discontinued if five, or more, lots are not accepted and all products from that source must be rejected. Importation and inspection should not resume until the responsible authority is satisfied that the producer has taken the necessary action to improve the quality of the submitted product. Tightened inspection should then be used as described above.

4.3 SINGLE SAMPLING PLANS FOR INSPECTION BY VARIABLES FOR PER CENT NONCONFORMING

(see ISO 3951: 1989 (E))

4.3.1 General

The principle of such sampling plans is presented in Section 2.5.1.2.

The application of ISO 3951 variables sampling plans may be summarised as follows:

Select the ‘s’ method (standard deviation unknown) or

the ‘σ‘ method (standard deviation is stable and known)

⬇

Set inspection level
(normal, tightened, reduced)

⬇

Set the AQL

⬇

Select sample size (n) & acceptability constant (k) and collect sample

⬇

Measure the characteristic x in each item in the sample

4.3.1.1 Decision rule for the ‘s’ method (see table 4)

(a) calculate the sample mean, $\bar{x}$, and

(b) calculate the estimated standard deviation, $s = \sqrt{\sum_{i=1}^{i=n} \frac{\left(x_i - \bar{x}\right)^2}{n-1}}$

(c) see Table 4.

4.3.1.2 Decision rules for the ‘σ‘ method (see table 3)

(This method should only be used when there is valid evidence that the standard deviation of the process can be considered constant and taken to be ‘σ‘. In this case, the controlling authorities shall check by any appropriate mean the relevance of the value of σ chosen by the professionnals)

a) calculate the mean of the sample $\bar{x}$

b) see Table 3

4.3.2 Recommended sampling plans by variables : s method

4.3.2.1 General

This section recommends the following simple sampling plans, for covering frequent inspection situations. They are extracted from the Standard ISO 3951, and are characterised by their AQL (of 0,65 % and 6,5 % for covering the most frequent cases), the size n of items in the sample and K the acceptance constant. Each plan is accompanied by a table which gives the probability of acceptance of the lots in function of the defective rate in these lots. For each AQL, a graph sums up the OC curves of the corresponding recommended plans.

The OC curves have been built point-by-point from the tables of values of ISO 3951.

Table 14 (from NMKL Procedure N°12, see reference 5) gives the number of items to be sampled at different lot sizes and inspection levels (normal inspection, tighten inspection and reduced inspection). It also gives the acceptability constant, K, at AQL’s of 0,65%, 2,5% and 6,5% respectively. Low AQL’s (0,65%) should be applied for critical defects while higher AQL should be applied for compositional parameters. Table 14 is a simplification of the “s-method” given in ISO 3951:1989.

TABLE 14: VARIABLE SAMPLING PLANS WITH UNKNOWN STANDARD DEVIATION

		Inspection level		
Lot size	n and k at AQLs (%)	Reduced	Normal	Tightened
2 - 8	n	**3**	**3**	**4**
	k at 0,65	1,45	1,65	1,88
	k at 2,5	0,958	1,12	1,34
	k at 6,5	0,566	0,765	1,01
9 - 15	n	**3**	**3**	**5**
	k at 0,65	1,45	1,65	1,88
	k at 2,5	0,958	1,12	1,40
	k at 6,5	0,566	0,765	1,07
16 - 25	n	**3**	**4**	**7**
	k at 0,65	1,45	1,65	1,88
	k at 2,5	0,958	1,17	1,50
	k at 6,5	0,566	0,814	1,15
26 - 50	n	**3**	**5**	**10**
	k at 0,65	1,45	1,65	1,98
	k at 2,5	0,958	1,24	1,58
	k at 6,5	0,566	0,874	1,23
51 - 90	n	**3**	**7**	**15**
	k at 0,65	1,45	1,75	2,06
	k at 2,5	0,958	1,33	1,65
	k at 6,5	0,566	0,955	1,30
91 - 150	n	**3**	**10**	**20**
	k at 0,65	1,45	1,84	2,11
	k at 2,5	0,958	1,41	1,69
	k at 6,5	0,566	1,03	1,33
151 - 280	n	**4**	**15**	**25**
	k at 0,65	1,45	1,91	2,14
	k at 2,5	1,01	1,47	1,72
	k at 6,5	0,617	1,09	1,35
281 - 500	n	**5**	**20**	**35**
	k at 0,65	1,53	1,96	2,18
	k at 2,5	1,07	1,51	1,76
	k at 6,5	0,675	1,12	1,39
501 – 1 200	n	**7**	**35**	**50**
	k at 0,65	1,62	2,03	2,22
	k at 2,5	1,15	1,57	1,80
	k at 6,5	0,755	1,18	1,42
1 201 – 1 320	n	**10**	**50**	**75**
	k at 0,65	1,72	2,08	2,27
	k at 2,5	1,23	1,61	1,84
	k at 6,5	0,828	1,21	1,46
1 321 - 10 000	n	**15**	**75**	**100**
	k at 0,65	1,79	2,12	2,29
	k at 2,5	1,30	1,65	1,86
	k at 6,5	0,886	1,24	1,48

Table 14 (continued)

10 001 - 35 000	n	**20**	**100**	**150**
	k at 0,65	1,82	2,14	2,33
	k at 2,5	1,33	1,67	1,89
	k at 6,5	0,917	1,26	1,51
35 001 - 150 000	n	**25**	**150**	**200**
	k at 0,65	1,85	2,18	2,33
	k at 2,5	1,35	1,70	1,89
	k at 6,5	0,936	1,29	1,51
150 001 - 500 000	n	**35**	**200**	**200**
	k at 0,65	1,89	2,18	2,33
	k at 2,5	1,39	1,70	1,89
	k at 6,5	0,969	1,29	1,51
500 001 and over	n	**50**	**200**	**200**
	k at 0,65	1,93	2,18	2,33
	k at 2,5	1,42	1,70	1,89
	k at 6,5	1,00	1,29	1,51

4.3.2.2 Sampling plans by variables (s-method), AQL = 0,65 % (see table 15 and figures 9 & 10)

Table 15: Probability of lot acceptance at AQL = 0,65 %, variable sampling plan (s-method)

Defective rates in the lots	Probability of lot acceptance Normal inspection plan			
	Letter-code D, AQL = 0,65%, n= 5, K =1,65 P_{95} [24]= 0,28% P_{50} [25]= 6,34% P_{10} [26] = 25,9%	Letter-code E, AQL = 0,65%, n= 7, K =1,75 P_{95} = 0,32% P_{50} = 4,83% P_{10} = 18,6%	Letter-code F, AQL = 0,65%, n= 10, K =1,84 P_{95} = 0,36% P_{50} = 3,77% P_{10} = 13,2%	Letter-code G, AQL = 0,65%, n= 15, K =1,91 P_{95} = 0,45% P_{50} = 3,09% P_{10} = 9,4%
0%	100%	100%	100%	100%
1%	96%	96%	97,5%	98%
2%	94%	94%	92,5%	95%
3%	86%	86%	86%	86%
4%	82%	82%	80%	78%
5%	78%	76%	73%	70%
6%	74%	70%	66%	62%
7%	69%	66%	59%	54%
8%	66%	60%	54%	46%
9%	61%	56%	48%	39%
10%	58%	52%	42%	34%
15%	42%	34%	23%	14%
20%	30%	21%	12%	5%
25%	23%	13%	6%	1,5%

[24] P_{95} = Rate of non-conforming items in lots accepted in 95% of cases

[25] P_{50} = Rate of non-conforming items in lots accepted in 50% of cases

[26] P_{10} = Rate of non-conforming items in lots accepted in 10% of cases

30%	15%	8%	2%	0%
35%	10%	5%	1%	0%
40%	6%	2%	0%	0%
45%	4%	1%	0%	0%
50%	2%	0%	0%	0%
100%	0%	0%	0%	0%

Table 15 (continued)

Defective rates in the lots	Probability of lot acceptance Normal inspection plan			
	Letter-code H, AQL = 0,65%, n= 20, K =1,96 P_{95} = 0,49% P_{50} = 2,69% P_{10} = 7,46%	Letter-code I, AQL = 0,65%, n= 25, K =1,98 P_{95} = 0,56% P_{50} = 2,53% P_{10} = 6,46%	Letter-code J, AQL = 0,65%, n= 35, K =2,03 P_{95} = 0,60% P_{50} = 2,21% P_{10} = 5,1%	Letter-code K, AQL = 0,65%, n= 50, K =2,08 P_{95} = 0,64% P_{50} = 1,94% P_{10} = 4,03%
0%	100%	100%	100%	100%
1%	84%	84%	84%	84%
2%	63%	62%	56%	48%
3%	44%	40%	32%	22%
4%	32%	28%	19%	10%
5%	24%	18%		4%
6%	16%	12%	6%	
7%	12%	8%	3,5%	1%
8%	8%	6%	2%	0,5%
9%	6%	4%	1%	
10%	4%	2%	0%	0%
15%	0%	0%	0%	0%

Figure 9: OC curve, variable sampling plan, s-method, AQL = 0,65 %, n = 5 to 15

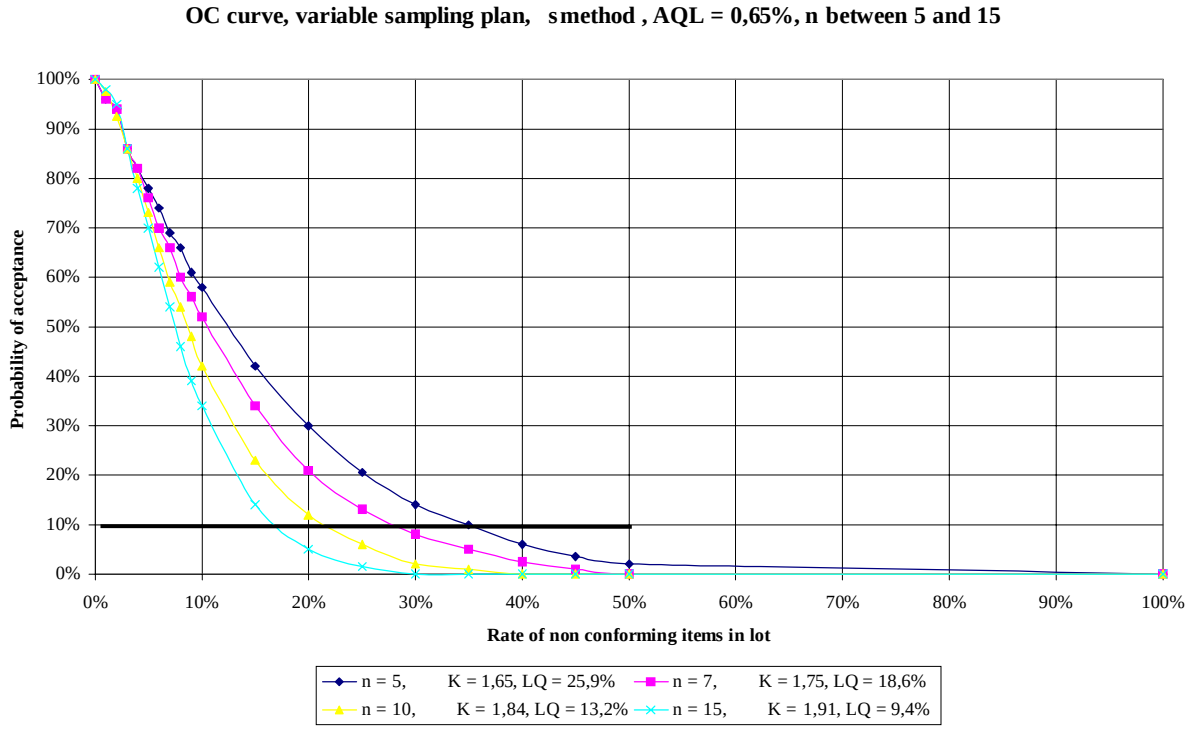
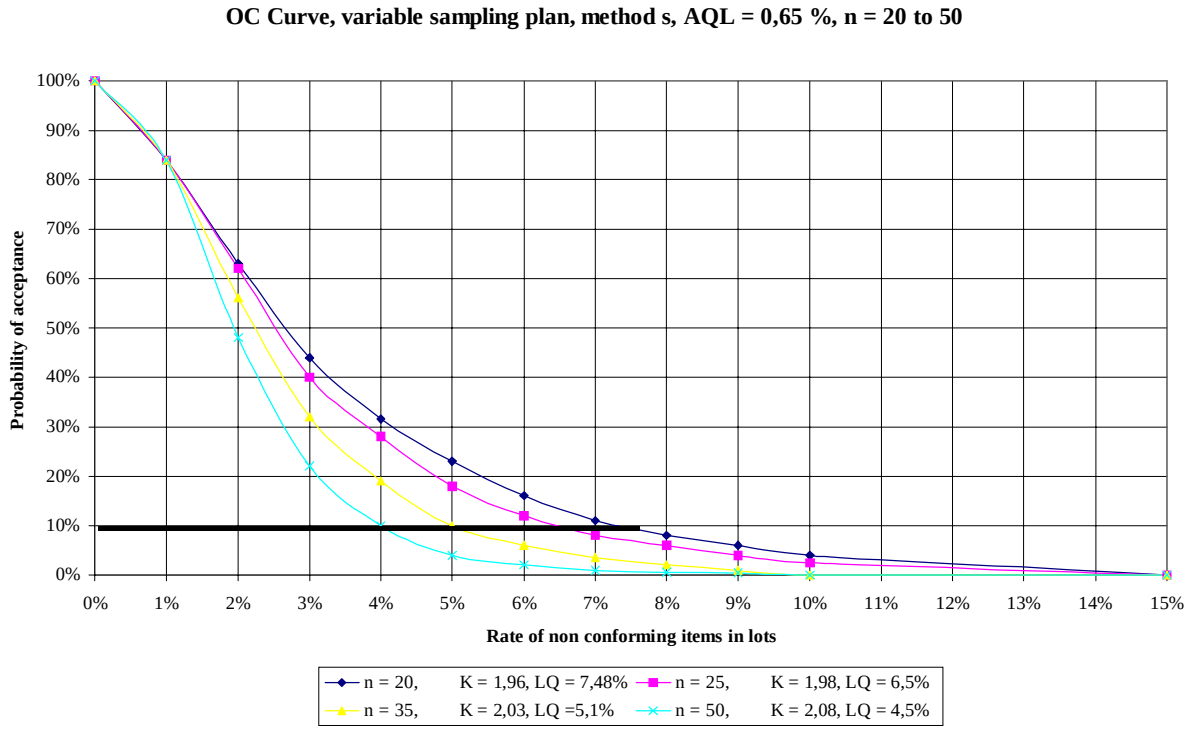


Figure 10: OC curve, variable sampling plan, s-method, AQL = 0,65 %, n = 20 to 50



4.3.2.3 Sampling plans by variables (s-method), AQL = 2,5% (see table 16, figures 11 and 12)

Table 16: Probability of lot acceptance, variable sampling plans (s-method), AQL = 2,5 %

Defective rates in the lots	Probability of lot acceptance Normal inspection plan			
	Letter-code D, AQL = 2,5%, n= 5, K =1,24 P_{95} = 1,38% P_{50} = 12,47% P_{10} = 35%	Letter-code E, AQL = 2,5%, n= 7, K =1,33 P_{95} = 1,5% P_{50} = 10,28% P_{10} = 27,4%	Letter-code F, AQL = 2,5%, n= 10, K =1,41 P_{95} = 1,61% P_{50} = 8,62% P_{10} = 21,4%	Letter-code G, AQL = 2,5%, n= 15, K =1,47 P_{95} = 1,91% P_{50} = 7,5% P_{10} = 16,8%
0%	100%	100%	100%	100%
1%	96%	96%	97,5%	99%
2%	94%	94%	92,5%	95%
3%	86%	86%	86%	86%
4%	82%	82%	80%	78%
5%	78%	76%	73%	70%
6%	74%	70%	66%	62%
7%	69%	66%	59%	54%
8%	66%	60%	54%	46%
9%	61%	56%	48%	39%
10%	58%	52%	42%	34%
15%	42%	34%	23%	14%
20%	30%	21%	12%	5%
25%	23%	13%	6%	1,5%
30%	15%	8%	2%	0%
40%	6%	2%	0%	0%
45%	4%	1%	0%	0%
50%	2%	0%	0%	0%
60%	0,5%	0%	0%	0%

Table 16 (continued)

Defective rates in the lots	Probability of lot acceptance Normal inspection plan			
	Letter-code H, AQL = 2,5%, n= 20, K =1,51 P_{95} = 2,07% P_{50} = 6,85% P_{10} = 14,2%	Letter-code I, AQL = 2,5%, n= 25, K =1,53 P_{95} = 2,23% P_{50} = 6,54% P_{10} = 12,8%	Letter-code J, AQL = 2,5%, n= 35, K =1,57 P_{95} = 2,38% P_{50} = 6% P_{10} = 10,9%	Letter-code K, AQL = 2,5%, n= 50, K =1,61 P_{95} = 2,51% P_{50} = 5,48% P_{10} = 8,7%
0%	100%	100%	100%	100%
1%	99%	99%	99%	99%
2%	95%	94%	94%	98%
3%	88%	88%	90%	90%
4%	78%	78%	75%	75%
5%	68%	66%	62%	58%
6%	58%	56%	50%	40%
7%	49%	44%	38%	28%
8%	40%	36%	25,5%	18%
9%	32%	28%	20%	11%
10%	26%	22,5%	14%	8%
12%	17%	12%	6%	2%
13%	13%	10%	4%	1%
14%	10%	7%	3%	0%
15%	8%	5%	0%	0%
20%	2%	1%	0%	0%
25%	0%	0%	0%	0%

Figure 11: OC curve, variable sampling plan, AQL = 2,5 %, n = 5 to 15

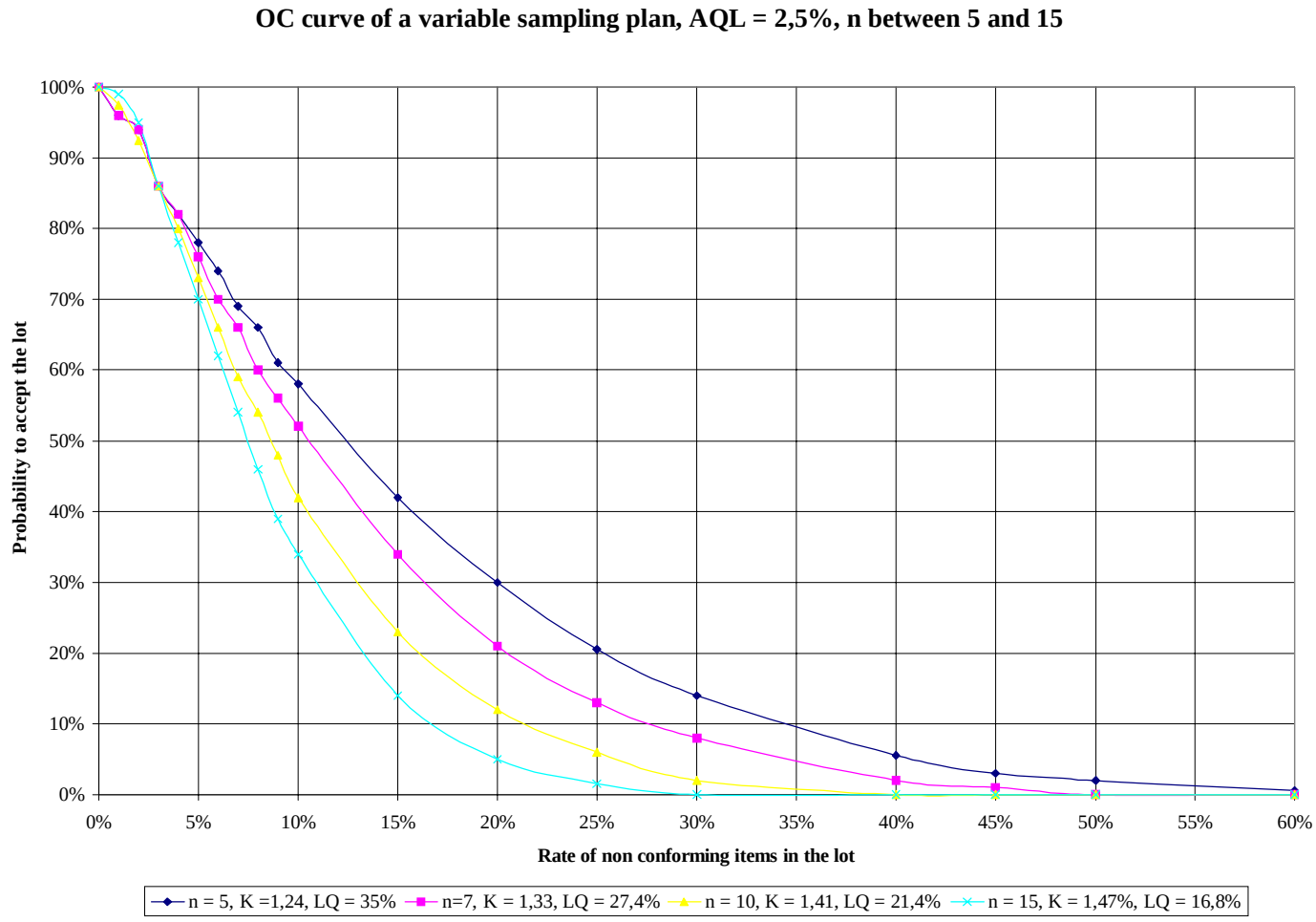
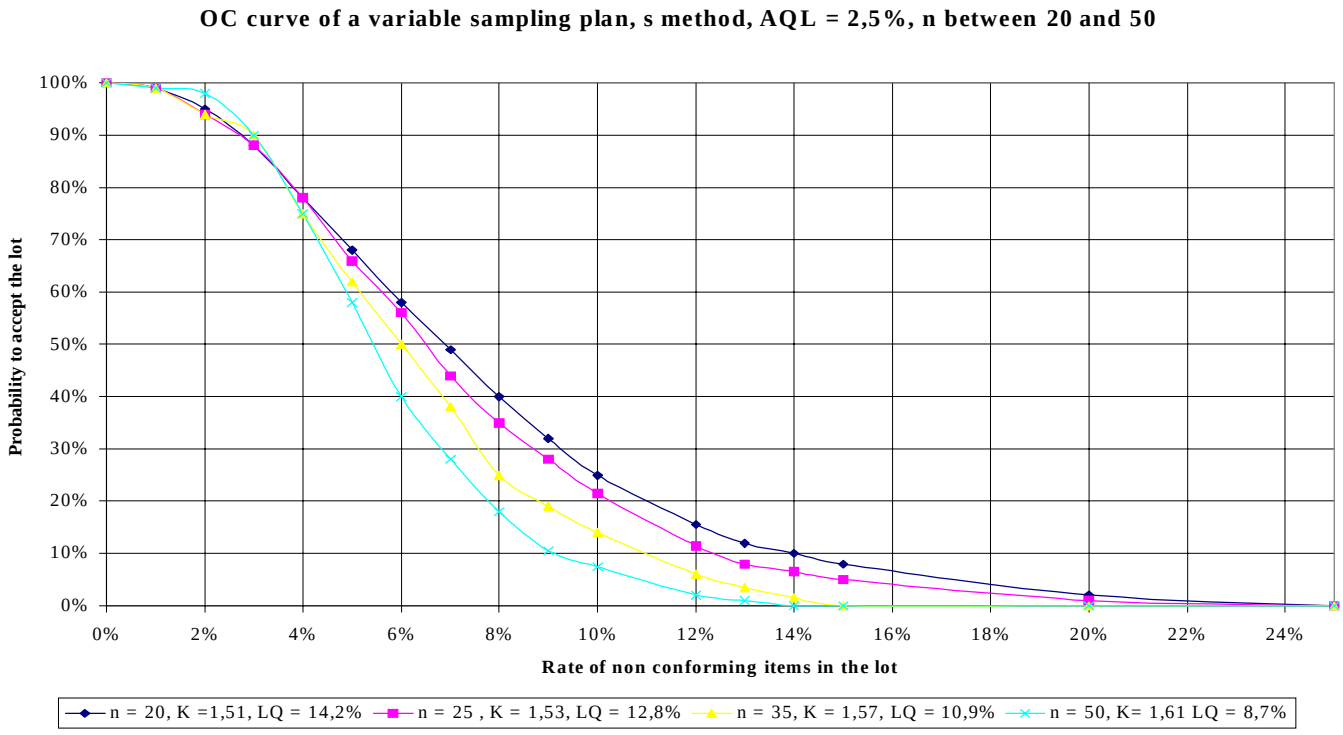


Figure 12: OC curve, variable sampling plan, AQL = 2,5 %, n = 20 to 50



4.3.3 Recommended sampling plans by variables : σ-method

4.3.3.1 General

This document recommends the following simple sampling plans, a for covering frequent inspetion situations. They are extracted from the Standard ISO 3951, and are characterised by their AQL (AQL of 0,65 % and 2,5 % covering the most frequent cases), the size n of items in the sample and K the acceptance constant. Each plan is accompanied by a table which gives the probability to accept the lots in function of the defective rate in these lots. For each AQL, a graph sums up the OC curves of the corresponding recommended plans.

The OC curves have been built point-by-point from the following .equation :

$$u_{PA} = \sqrt{n} \times (u_{1-p} - K)$$

where :

u_{PA} is the fractile of P_A order of the centered reduced normal law,

P_A is the probability to accept the lot having a defective rate of p

U_{1-p} is the fractile of 1-p order of the centered reduced normal law,

p is the the defective rate accepted in the lot with the probability P_A.

Table 17 (from NMKL N° 12, reference 5 and ISO 3951) indicates, for a normal inspection by variables (σ-method), the correspondence which is preferable for a better consumer protection (see clause 2.2.18) between the lot or batch size, the letter-code of the sample size, the sample size *n* and the acceptance constant K for given AQLs.

TABLE 17. VARIABLE SAMPLING PLANS WITH KNOWN STANDARD DEVIATION

		Inspection level		
Lot size	AQLs (%)	Reduced n/k	Normal n/k	Tightened n/k
2 - 8	0,65 2,5 6,5	**2** / 1,36 **2** / 0,936 **3** / 0,573	**2** / 1,58 **2** / 1,09 **3** / 0,755	**2** / 1,81 **2** / 1,25 **2** / 0,936
9 - 15	0,65 2,5 6,5	----‖----	----‖----	**2** / 1,81 **2** / 1,33 **3** / 1,01
16 - 25	0,65 2,5 6,5	----‖----	----‖----	**2** / 1,81 **3** / 1,44 **4** / 1,11
26 - 50	0,65 2,5 6,5	----‖----	**2** / 1,58 **3** / 1,17 **3** / 0,825	**3** / 1,91 **4** / 1,53 **5** / 1,20
51 - 90	0,65 2,5 6,5	----‖----	**3** / 1,69 **4** / 1,28 **5** / 0,919	**5** / 2,05 **6** / 1,62 **8** / 1,28
91 - 150	0,65 2,5 6,5	----‖----	**4** / 1,80 **5** / 1,39 **6** / 0,991	**6** / 2,08 **8** / 1,68 **10** / 1,31
151 - 280	0,65 2,5 6,5	----‖----	**5** / 1,88 **7** / 1,45 **9** / 1,07	**8** / 2,13 **10** / 1,70 **13** / 1,34
281 - 500	0,65 2,5 6,5	**2** / 1,42 **3** / 1,01 **4** / 0,641	**7** / 1,95 **9** / 1,49 **12** / 1,11	**10** / 2,16 **14** / 1,75 **18** / 1,38
501 - 1 200	0,65 2,5 6,5	**3** / 1,69 **4** / 1,11 **5** / 0,728	**8** / 1,96 **11** / 1,51 **15** / 1,13	**14** / 2,21 **19** / 1,79 **25** / 1,42
1 201 - 3 200	0,65 2,5 6,5	**4** / 1,69 **5** / 1,20 **7** / 0,797	**11** / 2,01 **15** / 1,56 **20** / 1,17	**21** / 2,27 **28** / 1,84 **36** / 1,46
1 320 - 10 000	0,65 2,5 6,5	**6** / 1,78 **8** / 1,28 **11** / 0,877	**16** / 2,07 **22** / 1,61 **29** / 1,21	**27** / 2,29 **36** / 1,86 **48** / 1,48
10 001 - 35 000	0,65 2,5 6,5	**7** / 1,80 **10** / 1,31 **14** / 0,906	**23** / 2,12 **32** / 1,65 **42** / 1,24	**40** / 2,33 **54** / 1,89 **70** / 1,51
35 001 - 150 000	0,65 2,5 6,5	**9** / 1,83 **13** / 1,34 **17** / 0,924	**30** / 2,14 **42** / 1,67 **55** / 1,26	**54** / 2,34 **71** / 1,89 **93** / 1,51
150 001 - 500 000	0,65 2,5 6,5	**12** / 1,88 **18** / 1,38 **24** / 0,964	**44** / 2,17 **61** / 1,69 **82** / 1,29	**54** / 2,34 **71** / 1,89 **93** / 1,51
500 001 and over	0,65 2,5 6,5	**17** / 1,93 **25** / 1,42 **33**/ 0,995	**59** / 2,18 **81** / 1,70 **109** / 1,29	**54** / 2,34 **71** / 1,89 **93** / 1,51

4.3.3.2 Sampling plans by variables (σ-method), AQL = 0,65 % (see table 18 and figures 13 and 14)

Table 18: Probability of lot acceptance, variable sampling plans, σ-method, AQL = 0,65 %

Defective rates in the lots	**Probability of lot acceptance Normal inspection plan**			
	Letter-code E, AQL = 0,65%, n= 3, K =1,69 P_{95} = 0,32% P_{50} =4,55% P_{10} = 18,6%	Letter-code F, AQL = 0,65%, n= 4, K =1,80 P_{95} = .0,36% P_{50} =3,6% P_{10} = 13,2%	Letter-code G, AQL = 0,65%, n= 5, K =1,88 P_{95} = 0,45% P_{50} =3% P_{10} = 9,41%	Letter-code H, AQL = 0,65%, n= 7, K =1,95 P_{95} = .0,49% P_{50} =2;56% P_{10} = 7,46%
0%	100%	100%	100%	100%
0,65%	91,5%	91,4%	91,2%	92,1%
1%	86,5%	85,4%	84%	84,1%
2%	73,5%	69,4%	65,1%	60,8%
3%	62,9%	56,4%	50%	42,7%
4%	54,2%	46,1%	38,6%	29,9%
5%	46,9%	37,8%	29,9%	20,9%
6%	40,7%	31,2%	23,3%	14,7%
7%	35,5%	25,8%	18,3%	10,4%
8%	31,1%	21,5%	14,4%	7,4%
9%	27,3%	17,9%	11,4%	5,3%
10%	24%	15%	9%	3,8%
15%	12,9%	15%	2,9%	0,8%
17 %	10%	4,5%	1,9%	0,4%
20%	7,1%	2,8%	1%	0%
25%	3,9%	1,2%	0,3%	0%
30%	2,2%	0,5%	0%	0%
35%	1,2%	0,2%	0%	0%
40%	0,6%	0,1%	0%	0%
45%	0,3%	0%	0%	0%
50%	0,2%	0%	0%	0%
60%	0%	0%	0%	0%

Table 18 (continued)

Defective rates in the lots	Probability of lot acceptance Normal inspection plan				
	Letter-code J, AQL = 0,65%, n= 11, K =2,01 P_{95} = 0,36% P_{50} =2,22% P_{10} = 5,1%	Letter-code K, AQL = 0,65%, n= 16, K =2,07 P_{95} = 0,64% P_{50} =1,92% P_{10} = 4,03%	Letter-code L, AQL = 0,65%, n= 23, K =2,12 P_{95} = 0,7% P_{50} =1,7% P_{10} = 3,24%	Letter-code M, AQL = 0,65%, n= 30, K =2,14 P_{95} = 0,74% P_{50} =1,6% P_{10} = 2,88%	Letter-code N, AQL = 0,65%, n= 44, K =2,17 P_{95} = 0,77% P_{50} =1,5% P_{10} = 2,36%
0%	100%	100%	100%	100%	100%
0,65%	94,2%	95,1%	95,6%	97%	98,1%
1%	85,3%	84,7%	83,4%	84,6%	85%
2%	55,8%	47,4%	37,8%	31,8%	22%
3%	33,4%	22,5%	13%	7,8%	2,8%
4%	19,5%	10%	4,1%	1,6%	0,3%
5%	11,3%	4,5%	1,3%	0,3%	0%
6%	6,5%	2%	0,4%	0,1%	0%
7%	3,8%	0,9%	0,1%	0%	0%
8%	2,2%	0,4%	0%	0%	0%
9%	1,3%	0,2%	0%	0%	0%
10%	0,8%	0,1%	0%	0%	0%
15%	0,1%	0%	0%	0%	0%
16%	0%	0%	0%	0%	0%

Figure 13: OC curve, variable sampling plan, σ-method, AQL = 0,65 %, n = 3 to 11

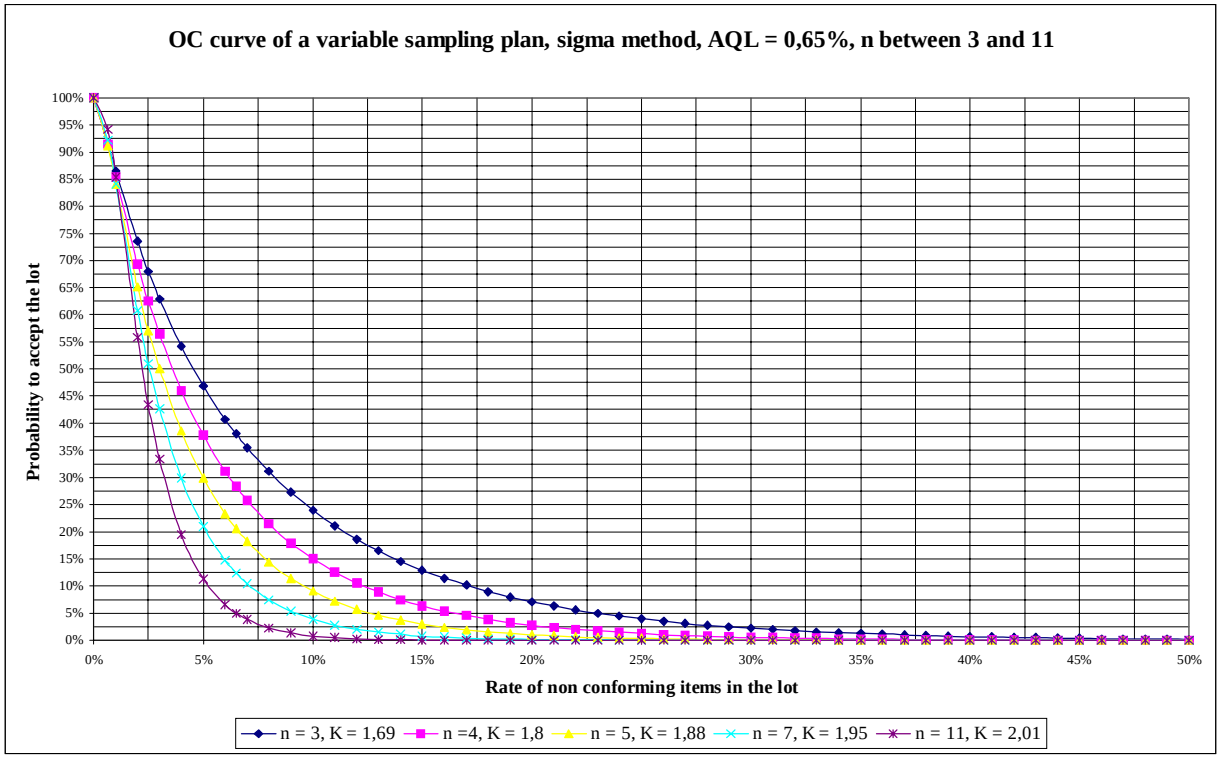
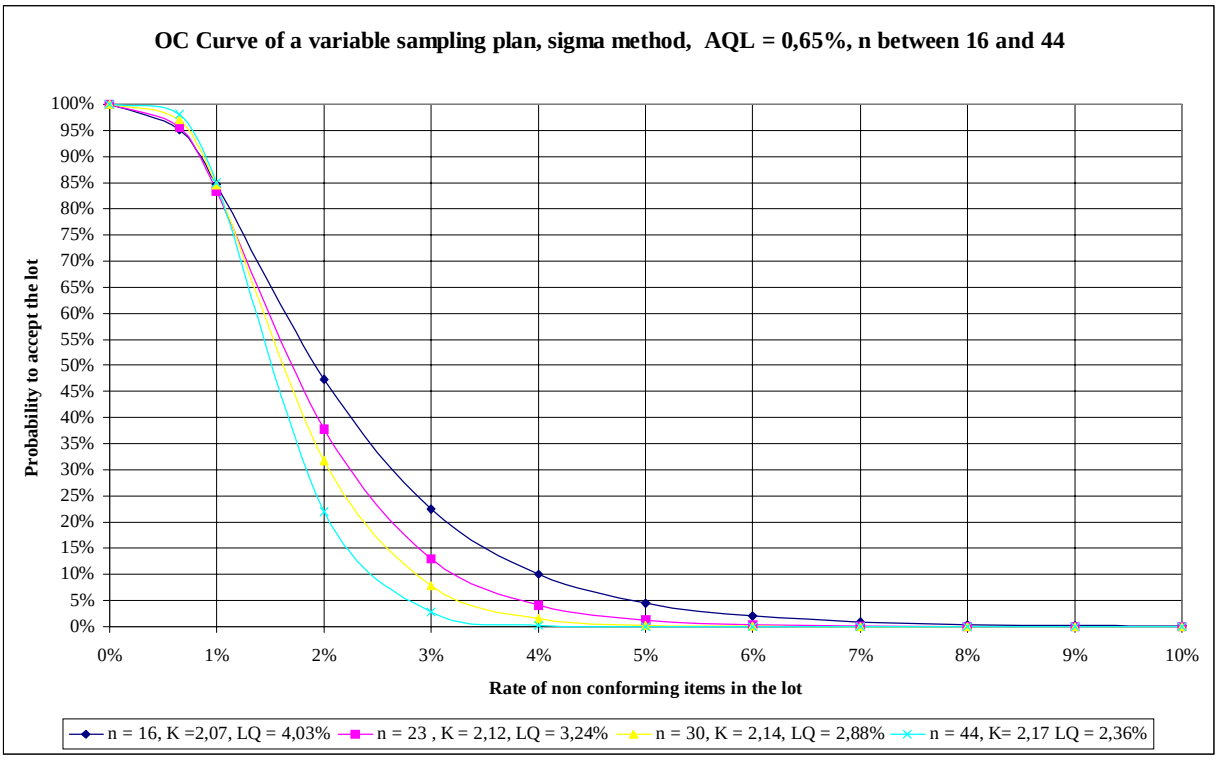


Figure 14: OC curve, variable sampling plan, σ-method, AQL = 0,65 %, n = 16 to 44



4.3.3.3 Sampling plans by variables (σ-method), AQL = 2,5 % (see Table 19 and figures 15 & 16)

Table 19: Probability of lot acceptance, variable sampling plans, σ-method, AQL = 2,5 %

Defective rates in the lots	Probability of lot acceptance Normal inspection plan				
	Letter-code D, AQL = 2,5%, n= 3, K =1,17 P_{95} = 1,38% P_{50} =12,1% P_{10} = 35%	Letter-code E, AQL = 2,5%, n= 4, K =1,28 P_{95} = 1,5% P_{50} =10% P_{10} = 27,4%	Letter-code F, AQL = 2,5%, n= 5, K =1,39 P_{95} = 1,65% P_{50} =8,23% P_{10} = 21,4%	Letter-code G, AQL = 2,5%, n= 7, K =1,45 P_{95} = 1,91% P_{50} =7,35% P_{10} = 16,8%	Letter-code H, AQL = 2,5%, n= 9, K =1,49 P_{95} = 2,07% P_{50} =6,81% P_{10} = 14,2%
0%	100%	100%	100%	100%	100%
1%	97,7%	98,2%	98,2%	99%	99,4%
2%	73,5%	93,9%	93,1%	94,5%	95,5%
3%	93,7%	88,5%	86,4%	87,3%	87,9%
4%	84,3%	82,7%	79%	78,7%	78,3%
5%	79,5%	76,7%	71,6%	69,7%	67,9%
6%	74,7%	70,9%	64,4%	60,9%	57,7%
7%	70,2%	65,2%	57,6%	52,7%	48,3%
8%	65,8%	59,9%	51,3%	45,3%	39,9%
10%	57,7%	50%	40,4%	32,8%	26,6%
15%	40,9%	31,3%	21,5%	13,7%	8,7%
20%	28,5%	19%	10%	5,4%	2,6%
25%	19,5%	11,3%	5,5%	2%	0,7%
30%	13,2%	6,5%	2,6%	0,7%	0,2%
35%	8,7%	3,7%	1,2%	0,2%	0%
40%	5,6%	2%	0,6%	0,1%	0%
45%	3,5%	1%	0,2%	0%	0%
50%	2,1%%	0,5%	0,1%	0%	0%
60%	0,7%	0,1%	0%	0%	0%
65%	0,4%	0%	0%	0%	0%
70%	0,2%	0%	0%	0%	0%
75%	0,1%	0%	0%	0%	0%
80%	0%	0%	0%	0%	0%
	0%	0%	0%	0%	0%

Table 19 (continued)

Defective rates in the lots	Probability of lot acceptance Normal inspection plan				
	Letter-code I, AQL = 2,5%, n= 11, K =1,51 P_{95} = 2,23% P_{50} =6,55% P_{10} = 12,8%	Letter-code J, AQL = 2,5%, n= 15, K =1,56 P_{95} = 2,38% P_{50} =5,94% P_{10} = 10,8%	Letter-code K, AQL = 2,5%, n= 22, K =1,61 P_{95} = 2,51% P_{50} =5,37% P_{10} = 9,23%	Letter-code L, AQL = 2,5%, n= 32, K =1,65 P_{95} = 2,62% P_{50} =5% P_{10} = 7,82%	Letter-code M, AQL = 2,5% n= 42, K =1,67 P_{95} = 2,73% P_{50} =4,75% P_{10} = 7,11%
0%	100%	100%	100%	100%	100%
1%	99,7%	99,9%	99,9%	99,9%	99,9%
2%	96,4%	97,2%	98,1%	98,3%	99,4%
3%	89,1%	89,3%	89,8%	90,4%	91,4%
4%	78,8%	77%	74,5%	71,6%	69,9%
5%	67,3%	62,9%	56,5%	50%	43,5%
6%	55,9%	49,2%	39,8%	29,5%	22,8%
7%	45%	37,2%	26,5%	16,2%	10%
8%	36,4%	27,4%	16,8%	8,3%	4,3%
9%	28,7%	19,8%	10,3%	4%	1,6%
10%	22,4%	14%	6,2%	1,9%	0,6%
11%	17,4%	10%	3,6%	0,8%	0,2%
13%	10%	4,7%	1,2%	0,2%	0%
15%	5,8%	2,1%	0,4%	0%	0%
20%	1,3%	0,3%	0%	0%	0%
25%	0,3%	0%	0%	0%	0%
30%	0,1%	0%	0%	0%	0%
31%	0%	0%	0%	0%	0%

Figure 15: OC curve, variable sampling plan, σ-method, AQL = 2,5 %, n = 3 to 9

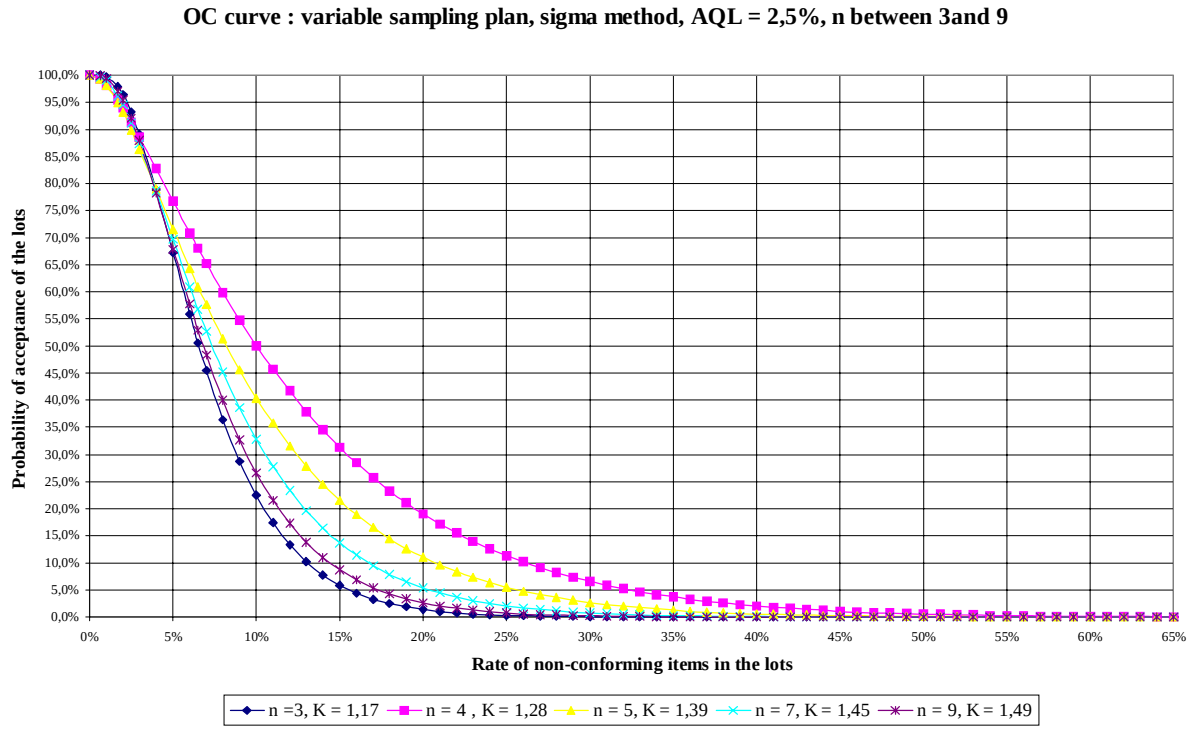
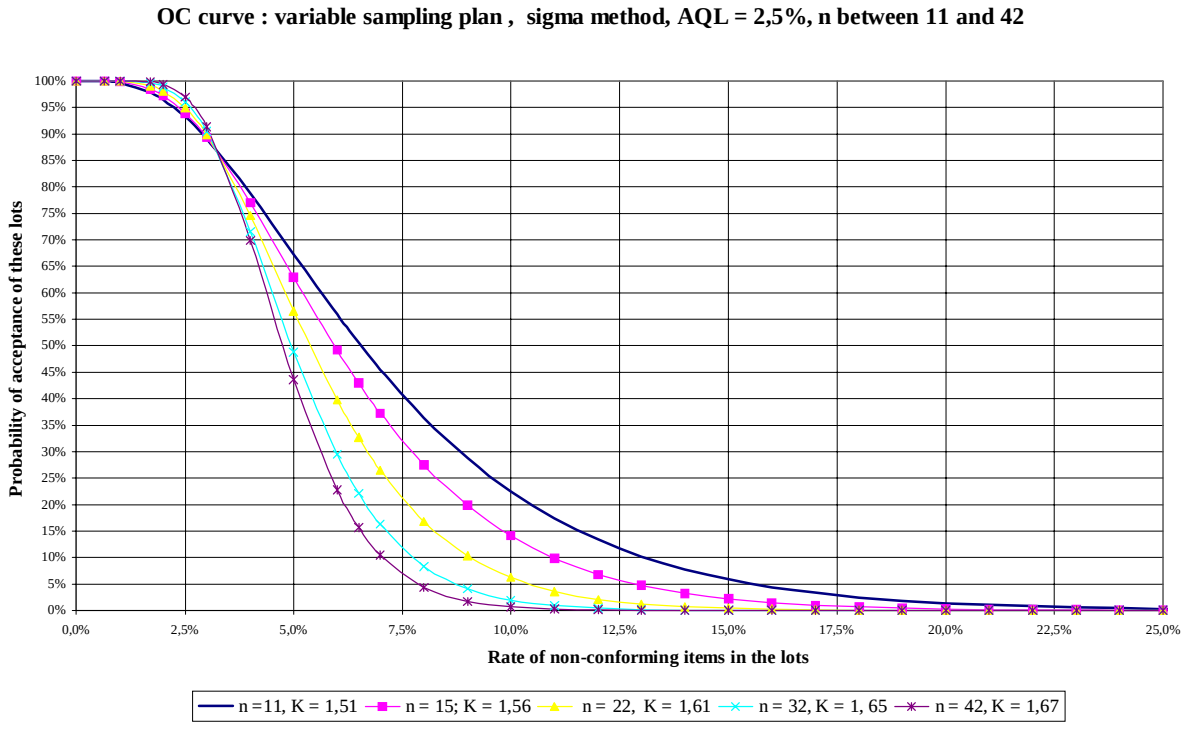


Figure 16: OC curve, variable sampling plan, σ-method, AQL = 2,5 %, n = 11 to 42



4.3.4 Rules and procedures of switching between inspection levels

(see article 19 of Standard ISO 3951)

When it is necessary, the switching towards a tightened inspection, which may lead to the rejection of the controlled lots, is mandatory. Nevertheless, the switching toward a reduced inspection, when the mean quality of a process is stable, at a level inferior to the AQL, is optional, at the discretion of the responsible authority. If there is sufficient proof, from the inspection tables, that the variability is in compliance with the statistical criteria, it can be envisaged to switch from the s method to the σ method, using the value of σ instead of s (see details in clause 2.2 and annex A of ISO 3951).

The switching of inspection level will of course imply a change of sampling plan (sample size, acceptance number).

The normal inspection is applied at the beginning of inspection (unless otherwise stated) and shall continue to be applied during inspection till a tightened inspection becomes necessary, or on the contrary, a reduced inspection becomes justified.

A tightened inspection shall be performed when 2 lots submitted to the original normal inspection are not accepted over 5 successive lots. The tightened inspection can be left when 5 successive lots at the first inspection have been accepted at the tightened inspection ; the normal inspection is then again performed.

It is possible to introduce a reduced inspection when 10 successive lots have been accepted at the normal inspection, under the following conditions :

a) these 10 lots would have been accepted if the AQL would have been fixed at the immediately inferior value to the one fixed by the plan (see Tables 2 and 3 of ISO 3951 : 1989);

b) the production is under statistical control;

c) the reduced inspection is considered as desirable by the users of the plans;

It is mandatory to stop the reduced inspection and to re-introduce a normal inspection if one of the following conditions are archived on lots at first inspection :

a) one lot is not accepted;

b) the production is delayed or erratic;

c) other conditions (change of supplier, of workers, of machines,…) imply the need to come back to a normal inspection.

4.4 SINGLE SAMPLING PLANS FOR AVERAGE CONTROL

4.4.1 Unknown standard deviation

Such a control is performed by using a test which aims at ensuring that, on average, the content of the controlled characteristic is at least equal to either the quantity given of the label of the product, or the quantity fixed by the regulation or a code of practice (e.g. net weight, net volume,…).

Description of the test

n is the sample size, in number of items, used for the test

$\bar{x} = \frac{\sum_{i=1}^{n} x_i}{n}$ is the sample mean of the n items in the sample

$$s = \sqrt{\frac{\sum_{i=1}^{n} \left(x_i - \bar{x}\right)^2}{n-1}}$$

is the standard deviation of the values of the items in the sample.

α is the significance level of the test, that is the probability of wrongly concluding that the mean content of the controlled chacteristic is less than the stated value when it is indeed greater than or equal to that value.

t_α is the value of the Student's t-distribution, on n-1 degrees of freedom, corresponding to the significance level α[27].

M is the stated value for the mean of the lot.

Decision Rules

The lot is accepted if:

$$\bar{x} \geq M - \frac{t_\alpha \times s}{\sqrt{n}}$$

and rejected otherwise.

The following Table provides t-values of the Student's distribution for some selected sample sizes and for α of 5 % and 0,5 %.

Number of Samples	t-value (α = 5%)	t-value (α = 0,5%)
5	2,13	4,60
10	1,83	3,25
15	1,76	2,98
20	1,73	2,86
25	1,71	2,80
30	1,70	2,76
35	1,69	2,73
40	1,68	2,71
45	1,68	2,69
50	1,68	2,68

4.4.2 Known standard deviation

Description of the test

n is the sample size, in number of items, used for the test

$$\bar{x} = \frac{\sum_{i=1}^{n} x_i}{n}$$ is the sample mean of the n items in the sample

σ is the known standard deviation.

α is the significance level of the test, that is the probability of wrongly concluding that the mean content of the controlled chacteristic is less than the stated value when it is indeed greater than or equal to that value.

u_α is the value of the Normal distribution, corresponding to the significance level α[28] ($u_{0,05}$ = 1,645, $u_{0,005}$ = 2,576).

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[27] α is generally taken at 5%, or 0,5%.

M is the stated value for the mean of the lot.

Decision Rules

The lot is accepted if:

$$\bar{x} \geq M - \frac{u_\alpha \times \sigma}{\sqrt{n}}$$

and rejected otherwise.

SECTION 5. THE SELECTION OF SAMPLING PLANS FOR THE INSPECTION BY VARIABLES OF BULK MATERIALS : KNOWN STANDARD DEVIATION

(see ISO/FDIS 10725 and ISO 11 648-1)

5.1 GENERAL

Normally, the sampling plans described in Section 5.1 should only be applied to a continuous series of lots from a single source. However, the plans described below may be utilised when data have been collected, describing the standard deviation of the quality characteristic, from isolated lots from a single source, over a prolonged period of time.

This draft standard addresses the need for sampling plans, by variables, for situations where the estimation of the lot mean of a single quality characteristic is the principal factor in the determination of lot acceptability. The sampling plans in this standard address the situations where a normal distribution of the quality characteristic occurs. However, users should not be too concerned about a deviation from normality, since the distribution of the sample grand average is usually very close to a normal distribution, unless the sample sizes are too small.

The standard may be applied:

- to *a continuing series of lots*
- to *lots in isolation* (*when the value of each standard deviation of the quality characteristic is considered to be known and stable;* for example, where a lot in isolation with respect to the purchaser may be part of a continuing series of lots produced by the supplier)
- when the specified quality characteristic χ is measurable on a continuous scale
- when the quality characteristic is stable, and the standard deviation known
- to a variety of bulk materials including liquids, solids (granular and powdered), emulsions and suspensions
- when a single specification limit is specified (however, under special circumstances, the standard is applicable when double specification limits are specified)

5.2 STANDARDISED SAMPLING PROCEDURES FOR THE INSPECTION OF INDIVIDUAL LOTS

The procedures involved in each step may be summarised as follows:

- **Selection of a sampling plan**

  The selection of a sampling plan involves the following steps, in particular for inspection of bulk material :

  ° the establishment of *standard deviations, costs, producer's risk quality, consumer's risk quality and discrimination distance (see definitions in 2.2.12)*

  If both the composite sample standard deviation (S_C) and the test sample standard deviation (S_T) control charts have no 'out of control' points, and if no other evidence gives doubt about their stability, it can be deemed that all standard deviations are stable. Methods for the confirmation and recalculation of standard deviations, including the utilisation of control charts, are provided in clause 12 of ISO/CD 10725-2.3

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[28] α is generally taken at 5%, or 0,5%.

° the specification of the *acceptance value(s)*

Acceptance value

When a lower specification limit is specified, the lower acceptance value is given by the equation:

$$\bar{x}_L = m_A - 0.562D$$

When an upper specification limit is specified, the upper acceptance value is given by the equation:

$$\bar{x}_U = m_A + 0.562D$$

where m_A is the producers' risk

D is the discrimination distance.

- **Drawing of increments from the lot**

An appropriate sampling device should be used together with representative sampling to afford n_i increments (i is the increment of rank i)

- **Preparation of one or more composite samples**

The n increments are pooled in order to produce n_c composite samples (A recommended, economical procedure is the preparation of *duplicate* samples by combining all odd numbered increments, to produce the first composite sample; and all even numbered increments, to produce the second composite sample.)

- **Preparation of test samples**

n_t test samples, of specified mass and particle size, are prepared from each composite sample, using appropriate crushing/grinding, sample division and mixing procedures.

- **Drawing of test portions for measurement**

n_m test portions, of specified mass, are drawn from each test sample

- **Measurement of specified quality characteristic of test portions**

A single measurement is performed on each test portion, to afford $n_c.n_t.n_m$ measurements per lot

- **Determination of lot acceptability**

The sample grand average ($\bar{x}$ **)** is calculated form the n_c composite sample averages (which are calculated from the n_T test sample averages which, themselves, are calculated from the n_M measurement results)

° When a single lower specification limit is specified:

Accept the lot if $\bar{x} \geq \bar{x}_L$

Reject the lot if $\bar{x} < \bar{x}_L$

° When a single upper specification limit is specified:

Accept the lot if $\bar{x} \leq \bar{x}_U$

Reject the lot if $\bar{x} > \bar{x}_U$

° When double specification limits are specified:

Accept the lot if $\bar{x}_L \leq \bar{x} \leq \bar{x}_U$

Reject the lot if either, $\bar{x} < \bar{x}_L$, or $\bar{x} > \bar{x}_U$

SECTION 6. REFERENCES

1. Micro-organisms in Foods. 2. Sampling for microbiological analysis: Principles and specific applications; International Commission on Microbiological Specifications for Foods, ICMSF, 1986, ISBN 0-632-015 67-5.

2. Cochran, WG : Sampling Techniques, 3rd Edition, Wiley, New York, 1977

3. Ducan, AJ : Quality Control and Industrial Statistics, 5th Edition, Irwin, Homewood, IL, 1986

4. Montgomery, DC : Introduction to Statistical Quality Control, 4th Edition, Wiley, New York, 2000

5. NMKL N° 12 : Guide on Sampling for Analysis of Foods, 2002

PROPOSED DRAFT GUIDELINES ON MEASUREMENT UNCERTAINTY

(At Step 5 of the Procedure)

Introduction

It is important and required by ISO/IEC 17025:1999 that analysts are aware of the uncertainty associated with each analytical result and estimates that uncertainty. The measurement uncertainty may be derived by a number of procedures. Food analysis laboratories are required, for Codex purposes, to be in control[1], use collaboratively tested methods when available[2], and verify their application before taking them into routine use. Such laboratories therefore have available to them a range of analytical data which can be used to estimate their measurement uncertainty.

Terminology

The accepted definition for Measurement Uncertainty is:

"Parameter, associated with the result of a measurement, that characterises the dispersion of the values that could reasonably be attributed to the measurand.

NOTES:

1. The parameter may be, for example, a standard deviation (or a given multiple of it), or the half-width of an interval having a stated level of confidence.
2. Uncertainty of measurement comprises, in general, many components. Some of these components may be evaluated from the statistical distribution of results of a series of measurements and can be characterised by experimental standard deviations. The other components, which can also be characterised by standard deviations, are evaluated from assumed probability distributions based on experience or other information.
3. It is understood that the result of a measurement is the best estimate of the value of a measurand, and that all components of uncertainty, including those arising from systematic effects, such as components associated with corrections and reference standards, contribute to the dispersion.

Recommendations

The following recommendations are made to governments:

1. For Codex purposes the term “measurement uncertainty” shall be used.
2. The measurement uncertainty associated with all analytical results is to be estimated and must, on request, be made available to the user (customer) of the results.
3. The measurement uncertainty of an analytical result may be estimated in a number of procedures, notably those described by ISO (1) and EURACHEM (2). These documents recommend procedures based on a component-by-component approach, method validation data, internal quality control data and proficiency test data. The need to undertake an estimation of the measurement uncertainty using the ISO component-by-component approach is not necessary if the other forms of data are available and used to estimate the uncertainty. In many cases the overall uncertainty may be determined by an inter-laboratory (collaborative) study by a number of laboratories and a number of matrices by the IUPAC/ISO/AOAC INTERNATIONAL (3) or by the ISO 5725 Protocols (4).

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[1] As outlined in Codex GL 27-1997 “Guidelines for the Assessment of the Competence of Testing Laboratories Involved in the Import and Export of Food"

[2] Where inter-laboratory studies are not possible, then a satisfactory surrogate for reproducibility such as the intra-laboratory reproducibility, or an approximation such as the Horwitz criterion, must be found

References

1. "Guide to the Expression of Uncertainty in Measurement", ISO, Geneva, 1993.

2. EURACHEM/CITAC Guide Quantifying Uncertainty In Analytical Measurement (Second Edition), EURACHEM Secretariat, BAM, Berlin, 2000. This is available as a free download from http://www.eurachem.ul.pt/

3. "Protocol for the Design, Conduct and Interpretation of Method Performance Studies", ed. W. Horwitz, *Pure Appl. Chem.*, 1995, 67, 331-343.

4. "Precision of Test Methods", Geneva, 1994, ISO 5725, Previous editions were issued in 1981 and 1986.

STATUS OF ENDORSEMENT OF METHODS OF ANALYSIS AND SAMPLING

A. Codex Committee on Fats and Oils
B. Codex Committee on Cocoa Products and Chocolate
C. Codex Committee on Milk and Milk Products
D. Codex Committee on Fish and Fishery Products
E. *Ad hoc* Intergovernmental Task Force on Fruit and Vegetable Juices
F. General Methods for the Detection of Irradiated Foods
G. Codex Committee on Food Additives and Additives and Contaminants
H. Codex Committee on Processed Fruits and Vegetables

A. CODEX COMMITTEE ON FATS AND OILS

Draft Standard for Fat Spreads and Blended Spreads (at Step 6)

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Fat Spreads and Blended Spreads	Lead	IUPAC 2.632, AOAC 994.02 or ISO 12193: 1994 (under revision) or AOCS Ca 18c-91.	Atomic absorption spectrophotometry (direct graphite furnace)		II	E
	Arsenic	AOAC 986.15	AAS	Subject to the finalization of provisions for Arsenic	II	TE
	Milk fat content	IUPAC 2.310, AOAC 990.27 or AOCS Ca 5c-87 (97)	Gravimetry followed by Gas Chromatography	CCFO to provide calculation as the method is for butyric acid	I	TE
	Vitamin A	AOAC 992.04	HPLC		II	E
	Vitamin D	AOAC 981.17	HPLC		II	E
	Vitamin E	IUPAC 2.432 or ISO 9936: 1997	HPLC		II	E

B. CODEX COMMITTEE ON COCOA PRODUCTS AND CHOCOLATE

Draft Standard for Chocolate and Chocolate Products (at Step 8)

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Chocolate and chocolate products	Milk Fat	IOCCC 5-1962 AOAC 945.34; 925.41B; 920.80	Titrimetry/Distillation		I	E
Chocolate and chocolate products	Non-cocoa butter vegetable fat		See below		I	E

1. Determination of centre and coating of filled chocolate

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

2. Determination of non-cocoa butter vegetable fat in chocolate and chocolate products

The following methods of analysis are the best available at the present time. Further systematic improvement is required. Documentation identifying the type of commercial blends of non-cocoa butter vegetable fats used must be made available upon request by competent authorities.

Detection of Non-Cocoa Butter Vegetable Fats in Chocolate

Detecting sterol breakdown products in refined vegetable fats added to chocolate by method AOCS Ce 10/02 (02).

Quantitative Determination of Non-Cocoa Butter Vegetable Fats[☂]

Determination of the triacyglycerols (C50, C52, C54) present in cocoa butters and non-cocoa butter vegetable fats by GC-FID in *J. Amer. Oil Chem. Soc.* (1980), **57**, 286-293. In milk chocolate, there is a need to correct for the milk fat

- **Interpretation:**

When type of non-cocoa butter vegetable fat is known, the amount of non-cocoa butter vegetable fat is calculated according to *J. Amer. Oil Chem. Soc.* (1980), **57**, 286-293.

When type of non-cocoa butter vegetable fat is not known, the calculation is made according to *J. Amer. Oil Chem. Soc.* (1982), **61 (3)**, 576-581.

☂ This method is intended to measure vegetable fats which are cocoa butter equivalents (CBE) i.e. SOS type triglycerides. Other vegetable fats can only be added in very limited amounts before they affect the physical properties of chocolate in a detrimental way. These can be determined by conventional methods i.e. fatty acid and triacyglycerol analyses.

C. CODEX COMMITTEE ON MILK AND MILK PRODUCTS

1. Methods of analysis referred back to CCMMP

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Fermented milks	Lactic acid	IDF 150:1991 ISO 11869:1997	Potentiometry, titration to pH 8.30	CCMMP should indicate whether the IDF method determines total acidity or lactic acid as in the provision		NE
		AOAC 937.05 AOAC 947.05	Spectrophotometry (for lactic acid in milk & milk products)	CCMMP should clarify what type method is requested since there cannot be two type II methods.	II	TE
	Microorganisms constituting the starter culture	IDF 149A:1997 (Annex A)	Colony count at 25°C, 30°C, 37°C and 45°C according to the starter organism in question	CCMMP should clarify whether a collaborative study has been performed and the type of the method.		NE
Yoghurt	*Streptococcus thermophilus* & *Lactobacillus delbrueckii* subsp. *Bulgaricus* >= 10^7 cfu/g	IDF 117B:1997 ISO 7889	Colony count at 37°C	Same question as above		NE
Yoghurt	*Streptococcus thermophilus* & *Lactobacillus delbrueckii* subsp. *bulgaricus* >= 10^7 cfu/g	IDF 146:1991 ISO 9232	Test for identification	Same question as above		NE

2. Methods of analysis proposed for standards under elaboration (advanced to Step 5 or 8)

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Cream and Prepared Creams	Milk protein	ISO 8968-1 |IDF20-1:2001 AOAC 991.20	Titrimetry (Kjeldahl)		I	E
Whey powders	Milk protein	ISO 8968-1 | IDF 20-1:2001 AOAC 991.20	Titrimetry (modified Kjeldahl)		I	E
	Water (not including water of crystallization of lactose)	IDF 26A:1993 AOAC 927.05	Gravimetry		I	E
Fermented milks	Protein	ISO 8968-1 | IDF 20-1:2001 AOAC 991.20	Titrimetry (Kjeldahl)		I	E
	Milk fat	ISO 1211:1999 IDF 1D:1996 AOAC 905.02	Gravimetry		I	E

3. Amendments to methods of analysis in adopted standards

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Cottage cheese	Milk fat in dry matter	IDF 126A:1988 ISO 8262-3:1987	Gravimetry (Weibull-Berntrop)		I	E
Individual cheeses	Dry matter (Total solids)	IDF 4A:1982 ISO 5534:1985 AOAC 926.08 applicable to all cheese	Gravimetry, drying at 102°C	CCMMP should clarify the difference in results with the previous method		NE

D. COMMITTEE ON FISH AND FISHERY PRODUCTS

Draft Standard for Boiled Dried Salted Anchovies (at Step 8)

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Boiled Dried Salted Anchovies	Sodium Chloride	AOAC 937.09	Titrimetry	Specification should be "chloride expressed as sodium chloride"	II	E
	Water Activity	AOAC 978.18		CCFFP to provide clarification as the method proposed applies to canned vegetables		NE
	Acid Insoluble Ash	Described in the Draft Standard		CCFFP should provide information on validation of the method		NE

E. *AD HOC* INTERGOVERNMENTAL TASK FORCE ON FRUIT AND VEGETABLE JUICES

See Agenda Item 6, para. 54.

F. GENERAL CODEX METHODS FOR THE DETECTION OF IRRADIATED FOODS

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Food containing crystalline sugar	Detection of irradiated food containing crystalline sugar	EN 13708:2001	ESR spectroscopy		II	E
Food containing silicate minerals	Detection of irradiated food	EN 13751:2002	Photostimulated luminescence		III	E
Herb, Species and Raw minced meat	Detection of irradiated food	EN 13783:2001 NMKL 137 (2002)	Direct Epifluorescent Filter Technique/Aerobic Plate Count (DEFT/APC)	Screening method	III	E
Food containing DNA	Detection of irradiated foodstuffs	EN 13784:2001	DNA comet assay	Screening method	III	E
Food containing silicate minerals	Detection of irradiated food containing silicate minerals	EN 1788 :2001	Thermoluminescence	EN 1788:1996 Updated	II	E

G. COMMITTEE ON FOOD ADDITIVES AND CONTAMINANTS

1) Food Additives

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Individual Foods[1]	Sulphites	EN 1988-1 : 1998-02 AOAC 990.28	Part 1: Optimized Monier-Williams method		III	E
Individual Foods[2]	Sulphites	EN 1988-2:1998 -02 NMKL 135 (1990)	Part 2: Enzymatic method		III	E
Table top sweeteners	Saccharin	EN 1376 : 1996-09 (confirmed 2001)	Spectrometric method		III	E
Table top sweeteners	Acesulfame K	EN 1377 : 1996-09 (confirmed 2001)	Spectrometric method		II	E
Table top sweeteners	Aspartame	EN 1378 : 1996-09 (confirmed 2001)	High performance liquid chromatography		II	E
Liquid table top sweeteners preparations	Cyclamate and Saccharin	EN 1379 : 1996-09 (confirmed 2001)	High performance liquid chromatography		II	E
All foods	Acesulfame K, Aspartame	EN 12856 : 1999-04	High performance liquid chromatography		II	E

[1] Hominy, fruit juice, sea food
[2] Wine, dried apples, lemon juice, potato flakes, sultanas, beer

	Saccharin	EN 12856 : 1999-04	High performance liquid chromatography		III[3]	E
All Foods	Cyclamate	EN 12857 : 1999-04	High performance liquid chromatography		II	E
	Cyclamate	NMKL 123 (1998)	Spectrophotometry	Previously endorsed as type II	III	E
All foods	Nitrates and/or Nitrites	EN 12014-1:1997-04	Part 1- General considerations			E
Meat Products	Nitrates and/or Nitrites	ENV 12014-3:1998-06 Part 3	Spectrometric determination of nitrate and nitrite content of meat products after enzymatic reduction of nitrate to nitrite		III[4]	TE
Meat Products	Nitrates and/or Nitrites	ENV 12014-4:1998-06 Part 4 NMKL 165 (2000)	Ion-exchange chromatographic method		III	TE

2) Contaminants

Cereal and Cereal Products	Ochratoxin A	EN ISO 15141-1:1998-10 Part 1	High performance liquid chromatographic method with silica gel clean up		II	E
		NMKL 143 (1997) EN ISO 15141-2:1998-10 Part 2	High performance liquid chromatographic method with bicarbonate clean up		III	E

[3] Method NMKL 122(1987) for Saccharin in Beverages and sweets endorsed as Type II (1997)

[4] Current methods for nitrites are AOAC 973.31 as Type II and ISO 2918.1975 as Type IV (To be re-validated and updated next year)

Cereals, shell-fruits and derived products (including peanuts)	Sum of aflatoxins B_1, B_2, G_1 and G_2	EN 12955 : 1999-07 ISO 16050[5]	HPLC with post column derivatization and immunoaffinity column clean up		III	E

3) Other Methods

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Foodstuffs	Nitrates/Nitrites	EN 12014-1:1997-04 Part 1: General considerations		These methods do not correspond to provisions under consideration in Codex Committees. Methods for pesticide residues are the responsibility of CCPR		NE
Vegetables and vegetable products	Nitrates/Nitrites	EN 12014-2:1997-04 Part 2	HPLC/IC			NE
		EN 12014-5:1997-04 Part 5	Enzymatic determination of nitrate content of vegetable-containing food for babies and infants			NE
		EN 12014-7:1998-06 Part 7	Continuous flow method for the determination of nitrate content of vegetables and vegetable products after cadmium reduction			NE
Fatty food	Pesticides and PCBs	EN 1528-1: 1996-10 (confirmed 2001) Part 1: General considerations				NE
		EN 1528-2: 1996-10 Part 2:	Extraction of fat, pesticides and PCBs and determination of fat content			
		EN 1528-3: 1996-10 Part 3	Clean-up methods			
		EN 1528-4: 1996-10 Part 4:	Determination, confirmatory tests, miscellaneous			
Maize	Fumonisins B_1 and B_2	EN 13585 : 2001 – 11	HPLC with solid phase extraction clean-up			NE

[5] To be published in 2003

Note: The current Codex methods for aflatoxins are as follows

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type
~~Maize(corn))~~	~~Aflatoxins, total~~	~~AOAC 979.18~~	~~Holaday-Velasco minicolumn~~	**Deleted as no provision exists**	~~II~~
Peanuts (intended for further processing)	Aflatoxins, total	AOAC 975.36	Romer minicolumn		III
Peanuts (intended for further processing)	Aflatoxins, total	AOAC 979.18	Holaday-Velasco minicolumn		III
Peanuts (raw)	Aflatoxins, total	AOAC 991.31	Immunoaffinity column (Aflatest)		II
Peanuts (raw)	Aflatoxins, total	AOAC 993.17	Thin layer chromatography		III

H. CODEX COMMITTEE ON PROCESSED FRUITS AND VEGETABLES

1) General Methods of Analysis for Processed Fruits and Vegetables

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Processed fruits and vegetables	Calcium	AOAC 968.31	Titrimetry	Replaces CAC/RM 38-1970	II	E
Processed fruits and vegetables (except pickled cucumbers)	Fill of containers	CAC/RM 46-1972	Weighing	Retain the current method Delete references to “metal containers” and refer to ISO 90.1:1986 for determination of water capacity in metal containers	I	E
Processed fruits and vegetables	Packing medium ≥ 10ºBrix Canned berry fruits (raspberry, strawberry)	AOAC 932.12 ISO 2173:1978	Refractometry		I	E

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Processed fruits and vegetables	Sodium chloride	ISO 3634:1979	Potentiometry	Provision should read: "chloride expressed as sodium chloride"	III	E
Processed fruits and vegetables	Determination of Drained Weight - Method I	AOAC 968.30	Weighing	Replaces CAC/RM 36-1970. The following changes are proposed to the AOAC method: - Revise Section 2.1 Specifications for Circular Sieves to read: If total quantity of contents is less than ~~1.5 kg. (3 lbs)~~ *1 kg. (2 lbs)* use a sieve. - Revise second sentence of Section 3. Procedure to read: Without shifting the contents, ~~so~~ incline the sieve *approximately 20º from the horizontal* to facilitate drainage - Insert new sentence at the end of the paragraph: "*This determination should be performed at 20ºC±5ºC .*" The instructions omit two important steps: (1) the weighing of the full container; and (2) the weighing of the dry empty container. Both weights are required to calculate the percentage drained weight (solid content) and/or the percent liquid The commodity committee should provide clarification on how sections 2.1 and 2.2 should be amended		NE
Processed fruits and vegetables	pH	AOAC 981.12 ISO 11289:1993	Potentiometry	The commodity committee should identify the provisions and the standards concerned and consider ISO 1842:1991 for processed fruits and vegetables		NE

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Processed fruits and vegetables	Sulphites	EN 1988-1 : 1998-02 AOAC 990.28	Optimized Monier-Williams method	General method for sulphites as endorsed in section G.1 above	III	E
Processed fruits and vegetables	Total solids	AOAC 920.151	Gravimetry		I	E

2) Methods of Analysis included in Draft Standards

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Aqueous Coconut Products	Moisture	Subtracting total solids from 100	Calculation			NE
Aqueous Coconut Products	Non-fat solids	Subtracting total fats from total solids	Calculation			NE
Aqueous Coconut Products	Total fats	AOAC 989.05, IDF/AOAC method to be checked	Ether extraction	This method applies to milk and the Committee should clarify whether it is applicable to coconut products		NE
Aqueous Coconut Products	Total solids	AOAC 990.20	Oven extraction	This method applies to milk and the Committee should clarify whether it is applicable to coconut products		NE
Canned Stone Fruits	Drained weight	AOAC 968.30 ISO:2173:1978	Gravimetry	General method for processed fruits and vegetables	I	E
Canned Stone Fruits	Soluble solids	AOAC 932.14C	Refractometry	General method for processed fruits and vegetables	I	E
Pickles	Benzoic acid	NMKL 103 (1984) AOAC 983.16	Gas Chromatography	The commodity Committee should consider more modern methods (LC method) such as NMKL124 (1997)	II	E

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Pickles	Lead	ISO 6633:1984	Flameless atomic absorption spectrophotometry		IV	TE
Pickles	Sorbate	NMKL 103 (1984) AOAC 983.16	Gas Chromatography	The commodity Committee should consider more modern methods (LC method) such as NMKL124 (1997)	II	E
Pickles	Sulphur Dioxide			See General Method for sulphites (Section G.1)		
Pickles	Tin ≤ 250.0 mg/kg	ISO 2447:1998	Spectrophotometry	The commodity Committee should consider using the General Codex Method AOAC 980.19 and clarify why this method is proposed		NE

It is proposed to delete the methods for the determination of **acidity, salt, and drained weight** as these provisions are not specified in the Draft Standard for Pickles.

PROPOSED DRAFT GUIDELINES FOR EVALUATING ACCEPTABLE METHODS OF ANALYSIS

(At Step 3 of the Procedure)

SCOPE

1. These guidelines provide a framework for evaluating acceptable methods of analysis.

2. These guidelines are intended to assist countries in the application of requirements for trade in foodstuffs in order to protect the consumer and to facilitate fair trade.

3. Laboratories involved in the evaluation must comply with Codex Guidelines CAC/GL 27 on the competence of testing laboratories involved in the import and export of foods.

4. If a method of analysis has been endorsed by Codex, then preference should be given to using that procedure.

REQUIREMENTS

5. Methods should be assessed against the following criteria by laboratories involved in the import and export control of foods:

- accuracy
- applicability (matrix, concentration range and preference given to 'general' methods)
- detection/determination limits if appropriate for the determination being considered
- linearity
- precision; repeatability intra-laboratory (within laboratory), reproducibility inter-laboratory (within laboratory and between laboratories), but generated from collaborative trial data rather than measurement uncertainty considerations
- recovery
- selectivity (interference effects etc.)
- sensitivity

6. Their definition and approach to their estimation are given below.

ACCURACY

Definition
(as a concept)

The closeness of agreement between the reported result and the accepted reference value.

Note:

The term accuracy, when applied to a set of test results, involves a combination of random components and a common systematic error or bias component. {ISO 3534-1} When the systematic error component must be arrived at by a process that includes random error, the random error component is increased by propagation of error considerations and is reduced by replication.

(as a statistic)

The closeness of agreement between a reported result and the accepted reference value. {ISO 3534-1}

Note:

Accuracy as a statistic applies to the single reported final test result; accuracy as a concept applies to single, replicate, or averaged value.

Estimation
Wherever possible the use of traceable reference materials should be used to determine the accuracy of the method of analysis used.

[Swedish proposal to CEN TC275 and WG10]

If certified reference materials are used during a method evaluation exercise then the mean determined value can be compared against the mean known value by calculation of the z-score.

$$z = \frac{\left(X_{found} - X_{certified}\right)}{\sqrt{\frac{\sigma^2_{found}}{n_{found}} + \frac{\sigma^2_{certified}}{n_{certified}}}}$$

or, if certified reference material standard deviation data are unavailable 95% confidence limit data may be used as an estimate of certified reference material standard deviation.

$$z = \frac{\left(X_{found} - X_{certified}\right)}{\sqrt{\frac{\sigma^2_{found}}{n_{found}} + \left(\frac{CI}{2}\right)^2}}$$

A z-score within the range$|z| \leq 2$ is deemed to be satisfactory.

APPLICABILITY

Definition
The analytes, matrices, and concentrations for which a method of analysis may be used satisfactorily to determine compliance with a Codex standard.

Note:

In addition to a statement of the range of capability of satisfactory performance for each factor, the statement of applicability (scope) may also include warnings as to known interference by other analytes, or inapplicability to certain matrices and situations.

Estimation

This should detail the analytes, matrices and concentrations for which the method of analysis may be used satisfactorily to determine compliance with a Codex standard. This may also include warnings as to known interference by other analytes, or inapplicability to certain matrices and situations. The Youden approach a fractional factorial approach, is commonly used to assess applicability/ruggedness.

DETECTION/DETERMINATION LIMITS

Definition: Detection Limit

The detection limit is conventionally defined as field blank + 3σ, where σ is the standard deviation of the field blank value signal (IUPAC definition).

However, an alternative definition which overcomes most of the objections to the above approach (i.e. the high variability at the limit of measurement can never be overcome) is to base it on the rounded value of the reproducibility relative standard deviation when it goes out of control (where 3 σ_R = 100%; σ_R = 33%, rounded to 50% because of the high variability). Such a value is directly related to the analyte and to the measurement system and is not based on the local measurement system.

Definition: Determination Limit

As for detection limit except that 6σ or 10σ is required rather than 3σ.

However, an alternative definition that corresponds to that proposed for the detection limit is to use σ_R = 25%. This value does not differ much from that assigned to the detection limit because the upper limit of the detection limit merges indistinguishably into the lower limit of the determination limit.

Estimation

Where measurements are made at low analyte or property levels, e.g. in trace analysis, it is important to know what is the lowest concentration of the analyte or property value that can be confidently detected by the method. The importance in determining this, and the problems associated with it, arise from the fact that the probability of detection does not suddenly change from zero to unity as some threshold is crossed. The problems have been investigated statistically in some detail and a range of decision criteria proposed.

For validation purposes it is normally sufficient to provide an indication of the level at which detection becomes problematic. For this purpose the "blank + 3*s*" approach will usually suffice. Where the work is in support of regulatory or specification compliance, a more exact approach such as that described by IUPAC and various others is likely to be appropriate. It is recommended that users quote whichever convention they have used when stating a detection limit.

Detection Limit (LOD) - Quick Reference	
What to analyse	**What to calculate from the data**
a) 10 independent sample blanks measured once each. or	*Sample standard deviation* 's' of a) sample blank values, or b) fortified sample blank values
b) 10 independent sample blanks fortified at lowest acceptable concentration measured once each	Express LoD as the analyte concentration corresponding to a) mean sample blank value + 3*s* or b) 0 + 3*s*
This approach assumes that a signal more than 3s above the sample blank value could only have arisen from the blank much less than 1% of the time, and therefore is likely to have arisen from something else, such as the measurand. Approach a) is only useful where the sample blank gives a non-zero standard deviation. Getting a true sample blank can be difficult.	
c) 10 independent sample blanks fortified at lowest acceptable concentration, measured once each	*Sample standard deviation 's'* of the fortified sample blank values Express LoD as the analyte concentration corresponding to sample blank value +4.65*s* (derives from hypothesis testing)
The 'lowest acceptable concentration' is taken to be the lowest concentration for which an acceptable degree of uncertainty can be achieved. Assumes a normal practice of evaluating sample and blank separately and correcting for the blank by subtracting the analyte concentration corresponding to the blank signal from the concentration corresponding to the sample signal. If measurements are made under repeatability conditions, this also gives a measure of the repeatability precision (Annex A, A20)	

The determination limit (LoQ) is strictly the lowest concentration of analyte that can be determined with an acceptable level of repeatability precision and trueness. It is also defined by various conventions to be the analyte concentration corresponding to the sample blank value plus 6 or 10 standard deviations of the blank mean.

Note: Neither LoD nor LoQ represent levels at which quantitation is impossible. It is simply that the size of the associated uncertainties approach comparability with the actual result in the region of the LoD.

Determination Limit (LoQ) – Quick Reference	
What to analyse	**What to calculate from the data**
a) 10 independent sample blanks measured once each. Getting a true sample blank can be difficult.	*Sample standard deviation 's'* of sample blank value. Express LoQ as the analyte concentration corresponding to the sample blank value plus either: i) 6*s*, or ii) 10*s*
b) Fortify aliquots of a sample blank at various analyte concentrations close to the LoD. Measure, once each, 10 independent replicates at each concentration level.	Calculate the standard deviation 's' of the analyte value at each concentration. Plot *s* against concentration and put assign a value to the LoQ by inspection. Express LoQ as the lowest analyte concentration which can be determined with an acceptable level of uncertainty.
Normally LoQ forms part of the study to determine working range. It should not be determined by extrapolation below the lowest concentration fortified blank. If measurements are made under repeatability conditions, a measure of the repeatability precision at this concentration is also obtained.	

LINEARITY

Definition

The ability of a method of analysis, within a certain range, to provide an instrumental response or results proportional to the quality of analyte to be determined in the laboratory sample. This proportionality is expressed by an a priori defined mathematical expression. The linearity limits are the experimental limits of concentrations between which a linear calibration model can be applied with a known confidence level (generally taken to be equal to 1%)."

Estimation

For any quantitative method, it is necessary to determine the range of analyte concentrations or property values over which the method may be applied. Note this refers to the range of concentrations or property values in the solutions actually measured rather than in the original samples. At the lower end of the concentration range the limiting factors are the values of the limits of detection and/or quantitation. At the upper end of the concentration range limitations will be imposed by various effects depending on the instrument response system.

Within the working range there may exist a linear response range. Within the linear range signal response will have a linear relationship to analyte concentration or property value. The extent of this range may be established during the evaluation of the working range. Note that regression calculations on their own are insufficient to establish linearity. To do this a visual inspection of the line and residuals may be sufficient; objective tests, such as 'goodness-of-fit' tests, are better still. In general linearity checks require points at at least 10 different concentrations/property values.

Evaluation of the working and linear ranges will also be useful for planning what degree of calibration is required when using the method on a day-to-day basis. It is advisable to investigate the variance across the working range Within the linear range, one calibration point may be sufficient, to establish the slope of the calibration line. Elsewhere in the working range, multi-point (preferably 6+) calibration will be necessary. The relationship of instrument response to concentration does not have to be perfectly linear for a method to be effective but the curve should be repeatable from day to day. Note that the working and linear range may be different for different matrices according to the effect of interferences arising from the matrix.

Working and Linear Range - Quick Reference			
Analyse	***Repeats***	**What to calculate from the data**	**Comments**
1. Blank plus reference materials or fortified sample blanks at various concentrations	1	Plot measurement response (y axis) against measurand concentration (x axis). Visually examine to identify approximate linear range and upper and lower boundaries of the working range.	Ideally the different concentrations should be prepared independently, and not from aliquots of the same master solution.
Need at least 6 concentrations plus blank		Then go to 2.	This will give visual confirmation of whether or not the working range is linear. This stage is necessary to test a working range, thought to be linear and where it is intended to use single point calibration.
2. Reference materials or fortified sample blanks at at least 6 different concentrations within the linear range	3	Plot measurement response (y axis against measurand concentration (x axis). Visually examine for outliers that may not be reflected in the regression. Calculate appropriate regression coefficient. Calculate and plot residual values (difference between actual y value and the y value predicted by the straight line, for each x value). Random distribution about the straight line confirms linearity. Systematic trends indicate non-linearity. Then go to 3.	It is unsafe to remove outliers without first checking using further determinations at nearby concentrations. If variance of replicates is proportional to concentration then use a weighted regression calculation rather than a non-weighted regression. In certain circumstances it may be better to try to fit a non-linear curve to the data. Functions higher than quadratic are generally not advised.
3. As for LoQ (b)		As for LoQ. LoQ effectively forms the lower end of the working range.	Work with successively lower concentrations until the accuracy and precision becomes unacceptable.

PRECISION CHARACTERISTICS

Definitions

The closeness of agreement between independent test results obtained under stipulated conditions {ISO 3534-1}

Notes: {ISO 3534-1}

1. Precision depends only on the distribution of random errors and does not relate to the true value or to the specified value.
2. The measure of precision is usually expressed in terms of imprecision and computed as a standard deviation of the test results. Less precision is reflected by a larger standard deviation.
3. “Independent test results” means results obtained in a manner not influenced by any previous result on the same or similar test object. Quantitative measures of precision depend critically on the stipulated conditions. Repeatability and reproducibility conditions are particular sets of extreme conditions.

Repeatability [Reproducibility]: Precision under repeatability [reproducibility] conditions. {ISO 3534-1}

Repeatability conditions: Conditions where test results are obtained with the same method on identical test items in the same laboratory by the same operator using the same equipment within short intervals of time. {ISO 3534-1}

Reproducibility conditions: Conditions where test results are obtained with the same method on identical test items in different laboratories with different operators using different equipment. {ISO 3534-1}

Note:

When different methods give test results that do not differ significantly, or when different methods are permitted by the design of the experiment, as in a proficiency study or a material-certification study for the establishment of a consensus value of a reference material, the term "reproducibility" may be applied to the resulting parameters. The conditions must be explicitly stated.

Repeatability [Reproducibility] standard deviation: The standard deviation of test results obtained under repeatability [reproducibility] conditions. {ISO 3534-1}

Notes: {ISO 3534-1}

1. It is a measure of the dispersion of the distribution of test results under repeatability [reproducibility] conditions.
2. Similarly "repeatability [reproducibility] variance" and "repeatability [reproducibility] coefficient of variation" could be defined and used as measures of the dispersion of test results under repeatability [reproducibility] conditions.

Repeatability [Reproducibility] limit: The value less than or equal to which the absolute difference between two test results obtained under repeatability [reproducibility] conditions may be expected to be with a probability of 95%. {ISO 3534-1}

Notes:

1. The symbol used is r [R]. {ISO 3534-1}
2. When examining two single test results obtained under repeatability [reproducibility] conditions, the comparison should be made with the repeatability [reproducibility] limit

$$r\,[R] = 2.8\, s_r[s_R].$$ {ISO 5725-6, 4.1.4}

3 When groups of measurements are used as the basis for the calculation of the repeatability [reproducibility] limits (now called the critical difference), more complicated formulae are required that are given in ISO 5725-6:1994, 4.2.1 and 4.2.2.

Estimation

The calculated repeatability and reproducibility values can be compared with existing methods and a comparison made. If these are satisfactory then the method can used as a validated method. If there is no method with which to compare the precision parameters then theoretical repeatability and reproducibility values can be calculated from the Horwitz equation for concentrations down to 120 µg/kg or the modified equation at levels less than 120 µg/kg and greater than 13.8%.

i.e.

$$\sigma = 0.22c \qquad if \;\; c < 1.2\times10^{-7}$$
$$\sigma = 0.02c^{0.8495} \qquad if \;\; 1.2\times10^{-7} \le c \le 0.138$$
$$\sigma = 0.01c^{0.5} \qquad if \;\; c > 0.138$$

Definition

Proportion of the amount of analyte present or added to the test material which is extracted and presented for measurement.

Estimation

Analytical methods do not always measure all of the analyte of interest present in the sample. Analytes may be present in a variety of forms in samples not all of interest to the analyst. The method may thus be deliberately designed to determine only a particular form of the analyte. However a failure to determine all of the analyte present may reflect an inherent problem in the method. Either way, it is necessary to assess the efficiency of the method in detecting all of the analyte present.

Because it is not usually known how much of a particular analyte is present in a test portion it is difficult to be certain how successful the method has been at extracting it from the matrix. One way to determine the efficiency of extraction is to spike test portions with the analyte at various concentrations, then extract the fortified test portions and measure the analyte concentration. The inherent problem with this is that analyte introduced in such a way will probably not be held as strongly as that which is naturally present in the test portion matrix and so the technique will give an unrealistically high impression of the extraction efficiency. It is however the most common way of determining recovery efficiency, and it is recognised as an acceptable way of doing so. However the drawback of the technique should be borne in mind. Alternatively it may be possible to carry out recovery studies on reference materials, if suitable materials are available. Provided these have been produced by characterisation of natural materials rather than by characterisation of synthetic materials into which the analyte has been spiked, then the recovery study should accurately represent the extraction of real test portions.

Recoveries - Quick Reference

Analyse	Repeats	What to calculate from the data	Comments
Matrix blanks or samples unfortified and fortified with the analyte of interest at a range of concentrations	6	Determine recovery of analyte at the various concentrations. Recovery (%) = (C1-C2)/C3 X 100 Where, C1 = concentration determined in fortified sample C2 = concentration determined in unfortified sample C3 = concentration of fortification	Fortified samples should be compared with the same sample unfortified to assess the net recovery of the fortification. Recoveries from fortified samples or matrix blanks will usually be better than real samples in which the analyte is more closely bound.
Certified reference materials (CRM)		Determine recovery of analyte relative to the certified value	Depending on how the CRM was produced and characterised, it may be possible to get >100% recovery.

SELECTIVITY

Definition

Selectivity is the extent to which a method can determine particular analyte(s) in mixtures or matrices without interferences from other components.

Selectivity is the recommended term in analytical chemistry to express the extent to which a particular method can determine analyte(s) in the presence of interferences from other components. Selectivity can be graded. The use of the term specificity for the same concept is to be discouraged as this often leads to confusion.

Estimation

Selectivity/specificity are measures that assess the reliability of measurements in the presence of interferences. The selectivity of a method is usually investigated by studying its ability to measure the analyte of interest in test portions to which specific interferences have been deliberately introduced (those thought likely to be present in samples). Where it's unclear whether or not interferences are already present, the selectivity of the method cab be investigated by studying its ability to measure the analyte compared to other independent methods/techniques.

Confirmation of identity and selectivity/specificity - Quick Reference

What you do	How many times	Calculate / determine	Comments
Analyse samples, and reference materials by candidate and other independent methods.	1	Use the results from the confirmatory techniques to assess the ability of the method to confirm analyte identity and its ability to measure the analyte in isolation from other interferences.	Decide how much supporting evidence is reasonably required to give sufficient reliability.
Analyse samples containing various suspected interferences in the presence of the analytes of interest.	1	Examine effect of interferences – does the presence of the interferent enhance or inhibit detection or quantification of the measurands.	If detection or quantitation is inhibited by the interferences, further method development will be required.

SENSITIVITY

Definition

Change in the response divided by the corresponding change in the concentration of a standard (calibration) curve; i.e., the slope, s_i, of the analytical calibration curve.

Note:

This term has been used for several other analytical applications, often referring to capability of detection, to the concentration giving 1% absorption in atomic absorption spectroscopy, and to ratio of found positives to known, true positives in immunological and microbiological tests. Such applications to analytical chemistry should be discouraged.

Notes: {IUPAC-1987}

1. A method is said to be sensitive if a small change in concentration, c, or quantity, q, causes a large change in the measure, x; that is, when the derivative dx/dc or dx/dq is large.
2. Although the signal s_i may vary with the magnitude of c_i or q_i, the slope, s_i, is usually constant over a reasonable range of concentrations. s_i may also be a function of the c or q of other analytes present in the sample.

Estimation

This is effectively the gradient of the response curve, i.e. the change in instrument response that corresponds to a change in analyte concentration. Where the response has been established as being linear with respect to concentration, i.e. within the linear range of the method, and the intercept of the response curve has been determined, sensitivity is a useful parameter to calculate and use in formulae for quantitation. Sensitivity is sometimes used to refer to limit of detection but this use is not generally recommended.

[Note: much of the detailed recommendations in Appendix VII have been taken from published texts, specifically:

AOAC-I Peer Verified Methods, Policies and procedures, 1993, AOAC International, 2200 Wilson Blvd., Suite 400, Arlington, Virginia 22201-3301, USA.

W. J. Youden; Steiner, E. H. 'Statistical Manual of the AOAC-Association of Official Analytical Chemists', AOAC-I, Washington DC, 1975, p35.

"The Fitness for Purpose of Analytical Methods: A Laboratory Guide to Method Validation and Related Topics" Eurachem Guide, 1998, http://www.eurachem.ul.pt/guides/valid.pdf.

Nomenclature in evaluation of analytical methods, including detection and quantification capabilities (IUPAC Recommendations 1995). *Pure & Appl. Chem*., 1995, **67**, 1699-1723.

Detection in Analytical Chemistry – Importance, Theory and Practice. L. A. Curries, ACS Symposium Series 361, American Chemical Society, Washington DC 1988. Various chapters are recommended, particularly Ch4 (Kirchmer, C. J.) and Ch 16 (Kurtz, D. A. *et al.*)

Analytical Methods Committee, "Recommendation for the Definition, Estimation and Use of the Detection Limit", *The Analyst*, 1987, **112**, 199-204.

"Evaluation of Analytical Methods used for Regulation of Foods and Drugs", W. Horwitz, *Anal. Chem.* 1982, 54 (1), 67A - 76A.

M. Thompson, *Analyst*, 2000, 125, 385-386.**]**