codex alimentarius commission

FOOD AND AGRICULTURE ORGANIZATION OF THE UNITED NATIONS

WORLD HEALTH ORGANIZATION

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ALINORM 04/27/23

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX ALIMENTARIUS COMMISSION

Twenty-seventh Session
Geneva, Switzerland, 28 June - 3 July 2004

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

Budapest, Hungary
8 – 12 March 2004

Note: This document incorporates Codex Circular Letter CL 2004/-8MAS

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CX 4/50.2 **CL 2004/8-MAS**
March 2004

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 25th Session of the Codex Committee on Methods of Analysis and Sampling (ALINORM 04/27/23)**

A. MATTERS FOR ADOPTION BY THE 27th SESSION OF THE CODEX ALIMENTARIUS COMMISSION

PROPOSED AMENDMENTS TO THE PROCEDURAL MANUAL

1. Inclusion of *General Criteria for the Selection of Single-Laboratory Validated Methods of Analysis* (para. 18, Appendix II)
2. Amendments to the *Analytical Terminology for Codex Use* (paras. 66-72, Appendix II)

METHODS OF ANALYSIS AND SAMPLING

3. Methods of Analysis in Commodity Standards at different steps (paras. 78-101, Appendix VI- Sections A to D)
4. General Methods of Analysis for Additives and Contaminants (paras. 102-106, Appendix VI - Section E)

PROPOSED DRAFT GUIDELINES AT STEP 8

5. Draft General Guidelines on Sampling (para. 25, Appendix III)
6. Draft Guidelines on Measurement Uncertainty (para. 37, Appendix IV)

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 May 2004.**

PROPOSED DRAFT GUIDELINES AT STEP 5

7. Proposed Draft Guidelines for Evaluating Acceptable Methods of Analysis (para. 54, 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, Viale delle Terme di Caracalla, 00100 Rome, Italy **before 15 May 2004**.

B. REQUEST FOR COMMENTS AND INFORMATION

7. The Use of Analytical Results: Sampling Plans, Relationship Between the Analytical Results, the Measurement Uncertainty, Recovery Factors and Provisions in Codex Standards (for inclusion in the Procedural Manual) (para. 135, Appendix VII)

Governments and international organizations wishing to submit comments 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@cfri.hu, **before 15 September 2004.**

8. Methods for the determination of dioxins and related compounds (para. 119)

Governments and international organizations are invited to provide proposals and information on the current methods used for determination of dioxins and related compounds to the Delegation of Germany (Dr. Hermann Broll, Bundesinstitut für Risikobewertung, Postfach 33 00 13, 14191 Berlin, Germany, fax: +49 1 888 412-3635, e-mail: h.broll@bfr.bund.de) with a copy to the Secretary, Joint FAO/WHO Food Standards Programme, FAO, Viale delle Terme di Caracalla, 00100 Rome, Italy, **before 15 September 2004**.

SUMMARY AND CONCLUSIONS

The summary and conclusions of the 25th 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 new *General Criteria for the Selection of Single-Laboratory Validated Methods of Analysis* in the Procedural Manual and the amendment of some definitions in the *Analytical Terminology for Codex Use* (paras. 18 and 72, Appendix II);
- endorsed several methods of analysis in commodity standards at different steps of the Procedure; and amended several general Codex methods for additives and contaminants (paras. 77-106, Appendix VI);
- advanced to Step 8 the Draft General Guidelines on Sampling (para. 25, Appendix III);
- advanced to Step 8 the Draft Guidelines on Measurement Uncertainty (para. 37, Appendix IV);
- advanced to Step 5 the Proposed Draft Guidelines for Evaluating Acceptable Methods of Analysis (para. 54, Appendix V)

Other Matters of Interest to the Commission

The Committee:

- agreed to ask for comments on the recommendations on the Use of the Analytical Results (for inclusion in the Procedural Manual) for consideration at the next session (para. 135, Appendix VII);
- agreed to return to Step 2/3 the Proposed Draft Guidelines for Settling Disputes on Analytical (Test) Results (para. 58);
- agreed to return to Step 2/3 the document on the Fitness-for-purpose Approach (para.. 65);
- agreed to proceed with the review of the current *Analytical Terminology for Codex Use* in the Procedural Manual (para. 76);
- agreed to consider criteria for methods of analysis for foods derived from biotechnology (para. 117) and methods for the determination of dioxins and PCBs at its next session (para. 119).

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INTRODUCTION

1) The Codex Committee on Methods of Analysis and Sampling held its Twenty-fifth Session in Budapest, Hungary, from 8 to 12 March 2004, by courtesy of the Government of Hungary. The Session was chaired by Professor Peter Biacs, Director-General of the Hungarian Food Safety Office and by the Vice-Chairperson, Prof. Pal Molnár, Head of Food Quality Department of the Central Food Research Institute (KEKI). The Session was attended by 123 delegates and observers representing 38 Member Countries, one Member Organization (EC) and 14 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 Ferenc Nyújtó, Deputy State Secretary of Ministry of Agriculture and Regional Development. Dr Nyújtó welcomed the participants and expressed that it was a great honour for Hungary to host the Codex Committee on Methods of Analysis and Sampling, as it had been doing for many years. He emphasised the increasing interest of members for this Committee whose work is of great importance to other Codex Committees. Dr Nyújtó stressed the adoption of a new Food Law by the Hungarian Parliament last year that dedicates a special chapter to Codex Alimentarius and establishes the National Codex Committee. Emphasizing the role of the Codex Alimentarius standards in assuring food safety and their importance for harmonization and international food trade, Dr Nyújtó wished the delegates all success in their work.

ADOPTION OF THE AGENDA (Agenda Item 1)

3) The delegation of the European Community presented CRD 5 on the division of competence between the European Community and its Member States according to Rule of Procedure II Paragraph 5 of the Codex Alimentarius Commission.

4) The Committee adopted the Provisional Agenda as presented in CX/MAS 04/1. It agreed to consider Agenda Item 5 and Agenda Item 6b) one after the other taking into account that both items related to the evaluation of methods of analysis.

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

5) The Committee noted that a number of matters referred by the 26th Session of the Codex Alimentarius Commission (CAC), and other Codex Committees were for information purposes or would be discussed in more detail under relevant Agenda items. It also noted that some issues such as the follow-up to the evaluation of Codex were still being considered by other Codex Committees. In addition the Committee considered matters referred as follows:

The use of analytical results: sampling, relationship between the analytical results, the measurement uncertainty, recovery factors and provisions in Codex standards

6) The Committee recalled that the paper on the above matters had been considered at its 24th session and with the aim to make recommendations regarding enforcement of Codex commodity standards especially when making decisions as to whether a lot is in compliance with Codex specifications. It was also recalled that this matter had been referred to the Codex Committee on Food Import and Export Inspection and Certification Systems (CCFICS) and Commodity Committees for their consideration and comments and that these comments were presented in document CX/MAS 04/2-Add.1.

7) The Delegation of the United Kingdom introduced the document and indicated that there was no common understanding between the member states on the use of uncertainty and recovery value when deciding whether the sample is in compliance or not. The Delegation pointed out that the guideline document on "The Use of Analytical results: Sampling, Relationship between the Analytical Results, the Measurement

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[1] CX/MAS 04/2, CX/MAS 04/2-Add.1 (Prepared by the United Kingdom, for consideration in conjunction with Matters Referred from Other Codex Committees on the Use of the Analytical Results), CRD 4 (comments of Phillippines).

Uncertainty, Recovery Factors and Provisions in Codex standards" (Annex I to CX/MAS 04/2-Add.1) had been prepared to ensure that the interpretation of sampling, measurement of uncertainty and recovery is harmonized across Codex and to facilitate the uniform interpretation of Codex standards.

8) Several delegations supported further development of this document as it addressed essential issues. However they noted that it had been distributed quite late, therefore there was not enough time to consider it with national experts, which was important to provide a scientific basis for the document.

9) Some delegations drew the attention of the Committee to the fact that it was necessary to clarify to whom the Guidelines were targeted: to Codex Committees or to governments as this was important for further development of the text. In this regard it was indicated that Codex had adopted the IUPAC Guidelines on recovery where much guidance for governments on recovery was already provided.

10) The Delegation of New Zealand indicated that the document should be improved especially on the description of sampling procedures and the basis for the sampling plan; and that the proposal for the handling of uncertainty was not the only way to proceed. Further clarification was also needed on the interpretation of the results, therefore additional work during the session was necessary to improve the text.

11) It was suggested to split this document into two parts: one dealing with sampling, and the other with uncertainty and recovery.

12) Some delegations indicated that the concept of recovery was quite complex, as it is estimated in chemical quantitative analysis, except for Type I methods and that the definition of recovery should be discussed very thoroughly in order to avoid future problems. It was suggested to clarify the use of significant figures and better separate the uncertainty arising from sampling from that arising from analysis, in order to avoid possible confusion.

13) The Committee agreed that an *Ad Hoc* Working Group should be convened under the Chairmanship of the United Kingdom[2] to redraft the Guidelines with the understanding that it would be used by Codex Commodity Committees. It also agreed to consider the redrafted Guidelines under Agenda Item 12 "Other Business and Future Work" (see also paras 128) to 0 .

Single laboratory validated methods of analysis

14) The Committee recalled that following the request of its 24th session, the Codex Committee on Pesticide Residues (CCPR) had proposed criteria of a general nature for the selection of single-laboratory validated methods of analysis, to be included in the Procedural Manual after the General Criteria in order to recognize that inter-laboratory validation of methods of analysis was not always available and applicable for multi-residue analysis purposes.

15) After some discussion regarding the applicability of criteria, the Committee concluded that the proposed criteria should be of a general nature and should be incorporated into the Procedural Manual. It therefore amended the first bullet (i) by deleting the specific reference to the CCPR Guideline on Good Laboratory Practice.

16) In bullet (ii), the Committee deleted the reference to "assurance" in quality system in order to be consistent with the decision of the 24th Session and clarified that the use of the method was embedded in a quality system in compliance with the ISO/IEC 17025 document. It also clarified that the principles of Good Laboratory Practice were those established by OECD.

17) The Committee amended the last bullet to indicate that the verification of results with other validated methods was applicable "where available".

18) The Committee agreed to forward the proposed General Criteria for Selection of Single-Laboratory Validated Methods of Analysis to the Committee on General Principles for endorsement and subsequent adoption by the 27th Session of the Commission and inclusion in the Codex Procedural Manual after the section on General Criteria (see Appendix II).

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[2] Austria, Brazil, Canada, Czech Republic, Egypt, Finland, Germany, Japan, Netherlands, New Zealand, United States

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

19) The Committee recalled that the Draft Guidelines had been adopted by the Commission at Step 5 and circulated for comments at Step 6. The Committee had also agreed at its last session that an *Ad hoc* Working Group would meet prior to the session in order to consider the comments and facilitate discussion in the Plenary Session.

20) The Delegation of France presented the revised version of the Draft Guidelines prepared by the Working Group (CRD 6) taking into account all the written comments submitted to the meeting. Most comments were of an editorial nature and some substantial amendments had been made to the text as follows.

21) Section 2.3.2 Employment of Authorized Sampling Officers had been amended in the light of the comments received. As regards Section 2.4 Estimation Error it had been agreed that the case where the analytical error is larger than one third of the sampling error would not be covered by the Guidelines. The Working Group had also revised section 4.4 Single Sampling Plans for Average Control.

22) The Committee agreed to delete the reference to "authorized" persons in section 2.3.2 as it was not clear who would authorize them to carry out sampling and this may create confusion. The Committee also agreed to clarify that Lot Size was defined by the number of items in Tables 10, 14 and 17.

23) Many delegations supported the advancement of the Draft Guidelines to Step 8 in order to provide guidance to governments. Some delegations, while supporting the adoption of the document, pointed out that further guidance was needed concerning the application of the sampling plan to specific commodities, in order to facilitate its use by governments. The Committee agreed that Commodity Committees should be encouraged to develop sampling plans for specific commodities, where necessary, on the basis of the general Guidelines.

24) The Committee expressed its appreciation to the Delegation of France and to the Working Group for their excellent work in the development and finalization of these Guidelines addressing complex issues that would provide important guidance to governments.

Status of the Draft General Guidelines on Sampling

25) The Committee agreed to advance the Draft Guidelines to Step 8 for adoption by the 27th Session of the Codex Alimentarius Commission (see Appendix III). It was agreed that the General Guidelines, when adopted, would replace the current Sampling Plans for Prepackaged Foods (AQL 6.5) (CODEX 233-1969).

DRAFT GUIDELINES ON MEASUREMENT UNCERTAINTY[4]

26) The Committee recalled that the draft Guidelines had been adopted by the Commission at Step 5 and circulated for comments at Step 6.

27) The Committee considered the Guidelines section by section and in addition to some editorial amendments made the following changes.

General comments

28) The Delegation of New Zealand drew the attention of the Committee to the fact that the draft Guidelines did not provide enough information on how measurement of uncertainty was to be used, especially in assessing compliance. It indicated that both Measurement Uncertainty and Sampling Plans were used to assess conformance to product specifications, therefore their roles had to be clarified. The Delegation therefore proposed areas where the Guidelines should be expanded.

Introduction

29) The Committee deleted the reference to footnote two as the text of this footnote was already covered by the relevant ISO document.

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[3] ALINORM 03/23, Appendix IV, CL 2003/29-MAS, CX/MAS 04/3 (comments of European Community, Finland, Hungary, Japan, New Zealand, United States), CRD 4 (comments of the Philippines), CRD 6 (revised version prepared by the *Ad Hoc* Working Group)

[4] ALINORM 03/23, Appendix V, CL 2003/29-MAS, CX/MAS 04/4 (comments of Finland, Ireland, Japan and New Zealand), CX/MAS 04/4-Add.1 (comments of Iran), CRD 4 (comments of the Phillippines)

30) The Committee had a long discussion regarding the content and where to place an amendment proposed by the Delegation of the United Kingdom as to how the quantitative analytical results are to be expressed and reported by analysts. The Committee agreed that the range of "a ± 2u" represented a 95% level of confidence, to be consistent with the recommendation referring to the level of confidence.

31) Some delegations were of the view that the amendment was formulated like a recommendation and therefore better fit to the recommendation section while others argued that it clarified the expression of analytical results and suggested to place it in the Introduction. The Committee agreed to put the proposed wording as second paragraph of the Introduction.

32) The Committee clarified that the Guidelines were applied only to quantitative analysis therefore added a third sentence in the Introduction to this effect.

Terminology

33) It was indicated that the intent of the third note was not clear therefore it could be deleted, however the Delegation of Ireland drew the attention of the Committee to the fact that the use of international definitions such as those presented in ISO documents should not be altered as it was very important for accreditation of laboratories.

34) It was clarified that the definition for Measurement Uncertainty referred to "international" instead of "accepted" and that notes were part of this definition therefore a quotation mark was placed at the end of the last note.

Recommendations

35) The Committee deleted the first recommendation regarding the applicability of the Guidelines as it was already clear from the Introduction that they are to be used by Governments. Recommendations were also rearranged in order to put them in a more logical sequence.

36) It was proposed to hold the decision on the Guidelines until the item on the Use of Analytical Results had been discussed, as it contained recommendations on how measurement of uncertainty is to be used. However the Committee noted that significant progress had been made on the development of the draft Guidelines on Measurement Uncertainty and that it provided valuable guidance to governments. The Committee noted that the other document was at a very early stage of development and that it was not clear enough what the outcome of these discussions might be at this moment (see paras 128) to 0135).

Status of the Draft Guidelines on Measurement Uncertainty

37) The Committee agreed to forward the above Guidelines as amended during the session to the 27th Session of the Commission to Step 8 for final adoption (see Appendix IV).

38) The Delegation of New Zealand expressed their reservation on this decision.

PROPOSED DRAFT GUIDELINES FOR EVALUATING ACCEPTABLE METHODS OF ANALYSIS (Agenda Item 5)[5]

39) The Committee recalled that the last session had considered Proposed Draft Guidelines intended for governments that proposed two possible approaches: the traditional approach assigning numerical values to specific parameters and the "fitness-for-purpose" approach. The Committee had agreed to circulate for comments at Step 3 the Proposed Draft Guidelines reflecting the traditional approach and to redraft the document on "fitness-for-purpose", for consideration under Agenda Item 6b). The Committee recalled that both documents were discussed separately as they were not at the same stage of development but that they had been initially intended to be part of the same Guidelines for Evaluating Acceptable Methods of Analysis. The Committee therefore agreed to discuss Agenda Item 6b) after Item 5 (see paras. 59) to 65).

40) The Committee discussed the text section by section and made the following amendments.

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5 ALINORM 03/23, Appendix VII, CL 2003/29-MAS, CX/MAS 04/5 (comments of Brazil), CRD 4 (comments of the Philippines)

Scope

41) The Committee agreed that in paragraph 3 laboratories "should comply with Codex Guidelines CAC/GL 27" (instead of "must") as guidelines should not be too prescriptive.

Requirements

42) The Committee agreed that methods should be assessed "as appropriate" against the criteria listed, since all methods might not need to be assessed according to all criteria, and the third bullet point on detection/determination limits was amended accordingly.

43) As regards precision (fifth bullet point), the Committee had an exchange of views on the need to clarify the text and agreed to retain only the reference to repeatability intra-laboratory and reproducibility inter-laboratory as the purpose of the section was to describe requirements for methods, not to establish definitions.

44) The Committee also noted that the initial text corresponded to the definition of "precision" in the Procedural Manual and agreed that the definition should be amended to delete the reference to measurement uncertainty considerations at it created confusion. The Committee agreed to consider the amendment to the definition under Agenda Item 7 that would address the review of terminology.

Accuracy

Estimation

45) The Committee agreed with the proposal of the Delegation of Ireland that reference material should be matrix matched and with a similar level of analyte and the first sentence was amended accordingly.

46) The Committee agreed to refer to z-value rather than z-score in order to avoid confusion with the terminology used by IUPAC and inserted a reference to NMKL Procedure No. 9 (2001) that had developed the calculation of z-value presented in the working document. An additional sentence was added to clarify the relation between the z-value, the reference value and the confidence interval in the second equation.

47) The Delegation of New Zealand expressed the view that the section should cover estimation only and that bias should not be used as a criterion to accept or reject a method since bias could be corrected. After some discussion, the Committee added a sentence to the effect that a z-value outside the range $|z| \leq 2$ indicates a significant bias and a bias correction should be made in this case.

Detection/Determination Limits

48) The Delegation of the Republic of Korea expressed the view that the reference to the acronym "LoQ" for Determination Limit created confusion, as this should refer to limit of quantification and that these two limits were not equivalent in its national regulations. Other delegations pointed out that "determination limit" was equivalent to "limit of quantification" in their national regulations. In order to avoid confusion, the Committee agreed to avoid the use of acronyms and to specify which limit was applied throughout the section.

Linearity

49) The Committee agreed that results should be proportionate to the quantity of the analyte, as the current text erroneously referred to "quality". As it was noted that the text reflected the current definition of "linearity" in the Procedural Manual, the Committee agreed to make the relevant correction when discussing terminology under Agenda Item 7.

Precision Characteristics

Estimation

50) The Delegation of New Zealand expressed its objection to the use of an acceptance criterion based on precision and proposed to delete the current text and to specify only that repeatability and reproducibility should be estimated by standard procedures, such as IUPAC 1987 Protocol. The Delegation of the United Kingdom indicated that the concept of comparison should be retained especially as one element of the criteria approach was the assessment of precision and even methods that had been collaboratively tested might have a large precision characteristic.

51) The Delegation of the Netherlands proposed to remove the second sentence of the paragraph indicating in which case the method could be used as a validated method in order to make the text less prescriptive.

52) The Delegation of the European Community supported the current text as it was important to retain the notion of comparison of repeatability and reproducibility values to decide whether methods could be used as validated methods.

53) The Committee could not come to a conclusion on this question and agreed that it would require further consideration at the next session.

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

54) The Committee agreed to advance the Proposed Draft Guidelines, as amended at the current session, for adoption at Step 5 by the 27th Session of the Codex Alimentarius Commission (see Appendix V).

CRITERIA FOR EVALUATING ACCEPTABLE METHODS OF ANALYSIS (Agenda Item 6)

PROPOSED DRAFT GUIDELINES FOR SETTLING DISPUTES OVER ANALYTICAL (TEST) RESULTS (Agenda Item 6a)

55) The Committee recalled that its last session had agreed that the delegation of France would develop Proposed Draft Guidelines to address disputes arising from differences in laboratory results, and that this new work had subsequently been approved by the Commission.

56) The Delegation of France informed the Committee that it had not been able to prepare the document as several related issues were still under discussion in the Committee and had not yet been resolved, such as sampling, measurement uncertainty and the use of recovery factors, the use of significant figures, the application of specifications to the lot or the unit. The Delegation recalled that it had prepared a document at the last session based on ISO 4529:2000 including a procedure for the declaration of conformity and that it had been considered too complex for practical use, but that guidelines in this area should follow a scientific approach.

57) Several delegations supported the development of guidance for governments in order to facilitate dispute settlements and pointed out that the document should be practical enough to be used by governments. The Committee agreed that the document should address only disputes related to analytical methodology and should not consider sampling issues.

Status of the Proposed Draft Guidelines for Settling Disputes over Analytical (Test) Results

58) The Committee agreed that the Delegation of France would prepare a new version of the Proposed Draft Guidelines for consideration by the next session.

CONSIDERATION OF THE FITNESS-FOR-PURPOSE APPROACH TO EVALUATING METHODS OF ANALYSIS (Agenda Item 6b)[6]

59) The Committee recalled that the last session had considered Proposed Draft Guidelines for Evaluating Acceptable Methods of Analysis proposing two possible approaches: the traditional approach and the ”fitness-for-purpose” approach. The Committee had agreed that the Proposed Draft Guidelines applying the traditional approach would be circulated at Step 3 (see Agenda Item 5) and that the Delegation of the United Kingdom would redraft the document on “fitness for purpose” with the assistance of a drafting group for further consideration. The Committee recalled that these documents were not at the same stage of development but that they had been initially intended to be part of the Guidelines for Evaluating Acceptable Methods of Analysis. The Committee therefore discussed Agenda Item 6b) after Item 5 (see paras 39) to 54).

60) The Delegation of the United Kingdom, while introducing the revised document, indicated that the fitness for purpose approach took all values into account by defining a fitness function as a single parameter. The document also defined the related uncertainty function, explained how the estimated characteristic function could be constructed from precision, and presented some examples of the application of this new procedure. The Delegation stressed the importance of considering this issue at the international level as it would affect regulations and was already applied or under development in some areas of EC food legislation.

61) The Committee expressed its appreciation to the Delegation of the United Kingdom for developing this comprehensive document that addressed complex issues of importance for the future work of the Committee.

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[6] CX/MAS 04/7, CRD 4 (comments of the Philippines), CRD 8 (comments of the United States)

62) The Delegation of the United States expressed the view that this was an interesting approach and that it had no objection in principle to the elaboration of the document but that it was premature to apply it for regulatory purposes, and that further discussion would be needed in this respect.

63) The Delegation of New Zealand expressed its reservations to the approach proposed and referred to its earlier objections to the application of acceptance criteria to bias and precision. The Delegation pointed out that the fitness function was quite arbitrary and that it would require a solid scientific basis, especially as it was likely to affect regulatory decisions. In particular, fitness for purpose should examine the effect of bias and measurement uncertainty on decisions made using results generated by the test method. The Delegation indicated that it had carried out studies in this area and was prepared to provide the results to the Committee for further consideration.

64) The Delegation of Germany expressed its reservations on this approach as all characteristics were subsumed into one function and questioned whether it adequately reflected the characteristics of the method. The Delegation of the Netherlands highlighted the relation between the issues under consideration and the recommendations in the Draft Guidelines on Measurement Uncertainty and supported further consideration of the fitness-for-purpose approach in view of its importance.

65) The Committee agreed that the delegation of the United Kingdom, in cooperation with interested delegations, would revise the document for further consideration at the next session.

REVIEW OF THE ANALYTICAL TERMINOLOGY FOR CODEX USE IN PROCEDURAL MANUAL (Agenda item 7)[7]

66) The Committee recalled that at its last session it was agreed to initiate the revision of the Definitions contained in the Codex Procedural Manual and that this was approved by the 26th Session of the Commission as new work. It also recalled that a Circular Letter has been distributed requesting comments on the Analytical Terminology for Codex Use.

General comments

67) Some delegations stressed the need to have only one set of harmonized definitions in Codex and to establish different definitions only in case of necessity. Analytical terminology should be clearly defined and justified otherwise there was a possibility to get different data and results. It was indicated that international definitions contained in ISO or IUPAC documents were being revised therefore it was necessary to have cross references in order to ensure consistency. The Delegation of the Republic of Korea proposed to define and describe terminology that is not yet defined and which is used for describing the meaning of a certain term.

Result

68) The Delegation of New Zealand suggested defining the "Test method" or "Method" as it could simplify further definition of "Result". However the Committee noted that the proposed definition was already consistent with the definition in other international documents therefore left it unchanged.

Specificity

69) The Delegation of Austria noted that the definition of "specificity " was quite similar to "selectivity" and that its use created some confusion especially as "specificity" defined in Codex did not include the words "of similar behaviour" which were necessary for gaining and quenching effects of matrix substances. The Committee was informed about recently published statistical approaches for estimation of selectivity, based on the IUPAC definition[8]. As "selectivity" was well defined in IUPAC, the Committee agreed to delete the definition of "specificity" and in future to refer only to "selectivity" as defined in IUPAC.

Accuracy (as a concept) and accuracy (as statistic)

70) The Committee noted that the section on accuracy was quite confusing as it contained two definitions: one used as a concept and the other as a statistic and that the definition on a statistic contained a

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7 CL 2003/43-MAS; CX/MAS 04/8 (comments of Cuba, France, Iran, United States of America); CX/MAS 04/8-Add.1 (comments of Japan, New Zealand); CRD 7 (comments of Austria); CRD 11 (comments of Brazil).

8 Anal. Bioanal. Chem (2003), 377: 1060 - 1060

second note which was not quite correct. It was also noted that this definition needed harmonization with other Codex Committees such as the Committee on Pesticide Residues and the Committee on Residues of Veterinary Drugs in Foods. The Committee agreed that there should be only one definition of "accuracy" and this definition should be the one defined by ISO 3534-1.

Trueness

71) The Committee noted that there was some inconsistency between the definitions of trueness in Codex and ISO and decided to include the second sentence of the note in the ISO definition.

72) The amended definitions are presented in Appendix II.

Approach to the Review of Terminology

73) The Committee had a discussion on the approach to the revision of terminology.

74) In order to simplify work, it was proposed to use only references and sources from international definitions; however the Committee noted that it would be difficult to accept this as definitions were so important that they should be fully spelt out in the Procedural Manual.

75) The Delegation of Finland drew the attention of the Committee to the fact that it was difficult to proceed with the revision of further definitions without having the opinion of international organizations working in the area of terminology. It noted that this matter was considered at the Inter-Agency Meeting (IAM) before the CCMAS and suggested that a paper containing the comparison of definitions should be prepared for consideration by the next session of the Committee. Many delegations supported this proposal especially as ongoing work on the revision of definitions was carried out by relevant international organizations and this would facilitate harmonization of definitions for Codex purposes.

76) The Committee agreed that a Circular Letter would be prepared asking member governments and interested international organizations their suggestions as to which definitions should be necessary for Codex purposes and which current Codex definitions should be amended. The Committee agreed that this Circular Letter should include the compendium prepared in 2002 by IAM and including all current appropriate definitions developed or being revised by relevant international organizations. It also agreed that the Delegation of the United States with the assistance of AOAC and other interested members and observers[9] should prepare a paper in the light of comments received, addressing the above issues and providing an analysis and recommendations/rationale for appropriate definitions to be used for Codex purposes, for consideration by the next session of the Committee.

ENDORSEMENT OF METHODS OF ANALYSIS PROVISIONS IN CODEX STANDARDS (Agenda Item 8)[10]

77) The report of the *Ad hoc* Working Group on Endorsement of Methods of Analysis (CRD 1) was presented by its Chair, Dr Roger Wood (United Kingdom). The Committee endorsed the methods proposed with the following amendments and comments.

Committee on Fats and Oils

78) The Observer from AOCS drew the attention of the Committee to the proposal from the Committee on Fats and Oils to delete the year of publication in the reference as this would simplify the updating and endorsement process. It was also noted that under ISO/IEC 17025: 1999, analysts were required to use the most updated version of methods of analysis. This proposal was supported by other observers, who indicated that when significant changes were made to a method, a new number was given to the method and this avoided any confusion.

79) Several delegations supported the inclusion of the year of publication in the method as this was an important reference for laboratories, especially for regulatory purposes. They expressed the view that it was not possible to endorse all future changes that might occur in a method without being informed of those changes, and that the list of methods should be reviewed regularly to ensure that all updates were taken into account. The Committee agreed to retain the year of publication at this stage and to consider this question further at its next session.

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[9] Austria, Brazil, the EC, AOCS

[10] CX/MAS 04/9, CX/MAS 04/9-Add.1, CX/MAS 04/9-Add.2 (coments of Canada), CRD 1 (report of the Working group on Endorsement), CRD3 (comments o AAC), CRD 10 (comments of Brazil)

Fat Spreads and Blended Fat Spreads

80) The Committee noted that the level of 3% milk fat was the essential composition factor used to differentiate fat spreads from blended spreads. The Committee recalled that the CCFO had recommended to convert the butyric acid concentration into milk fat concentration and to report the range in which the milk fat concentration of a sample would lie, in the absence of a single agreed factor. Some delegations pointed out that the question of the factor should be clarified by the CCFO and that the method could not be fully endorsed until this was resolved. It was suggested that reference be made to the figure used by the World Customs Organization for fat spreads, but some delegations noted that an average figure established for customs purposes would not solve a problem of interpretation of analytical results.

81) The Committee agreed to endorse temporarily the methods proposed as Type I pending the definition of a conversion factor by the Committee on Fats and Oils.

Olive Oils and Olive-Pomace Oils

82) The Delegations of Italy and Morocco expressed their reservations on the inclusion of the ISO 15788-2: 2003 method for stigmastadienes as it had not been considered by the Committee on Fats and Oils and was not used in the framework of the International Olive Oil Council. The Committee agreed to endorse temporarily this method and to forward it to the CCFO for consideration. All other methods were endorsed as proposed, with the editorial corrections proposed by the Delegation of Spain.

Named Vegetable Oils

83) The Committee endorsed the revised list of methods proposed by the CCFO, including the deletion of several IUPAC methods that are not any longer available.

Committee on Fish and Fishery Products

Determination of Fish Content in Quick Frozen Fish Sticks

84) The Delegation of Indonesia pointed out that the Interim Nitrogen Factors used for white fish to calculate fish content originated from temperate regions and the Committee noted that additional factors for other species could be put forward for consideration by the Committee on Fish and Fishery Products in order to refine further the methodology. The method was endorsed as proposed.

***Ad hoc* Intergovernmental Task Force on Fruit and Vegetable Juices**

Fruit Juices and Nectars

85) The Committee noted that the Working Group on endorsement had agreed to consider for endorsement the methods corresponding to permitted ingredients and additives or processing aids, as listed in the Draft Standard (Annex – Section C.1), and that the other methods listed by the Task Force as quality methods or authenticity methods would not be considered for endorsement but listed separately.

86) The Delegation of Canada (Mrs Carla Barry, Chair of the WG on methods of analysis in the Task Force) recalled that the Task Force had agreed to define a complete set of methods as they were necessary as references at the international level but that it had not been possible at that stage to define values for the parameters related to authenticity and quality. These values might be established in the future and that would be facilitated by the harmonisation of methods of analysis. The Delegation therefore proposed that the Committee endorse the methods for authenticity and quality (section C.2 of CRD 1). This position was supported by the Delegation of Brazil, referring to its written comments, and several other delegations.

87) The Secretariat recalled that methods should correspond to specific provisions in standards and that so far the Committee had endorsed only methods for analytes or properties that were identified in Codex standards. If this approach was changed for fruit juices, this might also affect the overall endorsement process.

88) Some delegations pointed out that the provision in the *General Criteria of the Selection of Methods of Analysis* on "direct pertinence to the Codex standard" did not mean that a numerical value should be specified in the standard. The Secretariat referred to the "Recommendations for a Checklist of Information Required to Evaluate Methods of Analysis Submitted to the CCMAS for Endorsement" especially point *1.1.3 Analyte or Property* and *1.1.4 Codex Specification or Limit* requiring "the specification, limit, tolerance or guideline which is given in the standard and which provides the boundary between acceptable and unacceptable material" and noted that this boundary should be defined in the standard, whether it was numerical or not. Some delegations expressed the view that the *Recommendations* were not relevant as they

were not part of the Procedural Manual and the Committee agreed that they should not be taken into account in the case of fruit juices.

89) Some delegations proposed to endorse the methods only temporarily as Type IV in order to retain the valuable information resulting from the work of the Task Force, with the understanding that they would be endorsed when the relevant provisions were completed. The Delegation of Brazil pointed out that all the methods proposed had been validated and therefore should not be listed as Type IV. The Committee therefore agreed that the methods would be listed without a type as "temporarily endorsed" pending the establishment of numerical values by the Task Force.

90) The Committee made some corrections to the methods proposed for endorsement by the Working Group (section C.1 of CRD 1) and agreed to include methods for total nitrogen and for cellobiose to the list. The Committee noted that the ISO method for the determination of ascorbic acid was not validated and agreed that it should be retained as Type IV. The Delegation of Switzerland proposed to include method EN 14130 for the determination of Vitamin C but this was not accepted as this method had not been collaboratively tested.

91) The Delegation of Japan proposed that the applicability of methods for food additives that are also intrinsic constituents of fruit juices and nectars should be clarified. The Committee agreed that the Task Force should provide clarification in this respect.

Committee on Nutrition and Foods for Special Dietary Uses

Gluten Free Foods

92) The Committee had an extensive discussion on the proposed Enzyme-Linked Immunoassay R5 Mendez (ELISA) Method forwarded by the CCNFSDU for the determination of gluten. The Observer of the WGPAT (Prolamin Working Group) pointed out that method AOAC 991.19 was inadequate, especially as regards specificity and sensitivity and that the new method proposed was the only one that could determine all gliadin fractions, and the toxic gliadin peptide. The method had been collaboratively tested, the results had been published in scientific literature and all relevant information was available to analysts. In addition, only the extraction cocktail was patented but its composition was described in literature and could be used by laboratories. The Observer referred to the documents on the characteristics of the methods that had been presented to the Working Group and proposed to endorse the method as it represented a significant progress in order to address an important health problem.

93) The Observer of AOAC indicated that the colorimetric AOAC 991.19 method had been approved for final action in 2001, was collaboratively tested and publicly available and routinely used by laboratories.

94) The Delegation of Sweden expressed its concern with the fact that the proposed method was not publicly available to analysts and proposed to endorse the AOAC method for the determination of gluten as it was currently used in several countries for the analysis and control of gluten-free foods.

95) The Delegation of France pointed out that the ELISA method was not applicable to certain foods and questioned its relevance in a standard covering all gluten free foods, and drew the attention of the Committee to the comments of AAC in CRD 3, especially as regards the applicability to wheat starch hydrolysates.

96) Some delegations pointed out that the standard did not define clearly what should be measured since section 6.2 referred to a detection limit of 10 ppm without specifying whether this applied to gluten or gliadins. The Committee agreed to ask the CCNFSDU to clarify the applicability of the method and how it related to the provisions for "gluten free" in the standard.

97) The Delegation of the EC (Institute for Reference Material and Measurements) informed the Committee about its work to develop the European gliadin reference material, that was in the process of certification and indicated that the IRMM would initiate a comparison of methods at the end of 2004.

98) The Observer form AOECS expressed the view that it was essential to have a reference method to determine gluten in order to address the health problem faced by coeliac patients, and pointed out that a wide variability existed in the results obtained with the AOAC method. The Observer recalled that no method had been established in present Standard for Gluten Foods, and that the revision of the standard had been underway for several years, since there was no agreement at the moment on the levels or the method of determination.

99) The Observer from AOCS proposed that the R5 ELISA method should be considered by one of the IAM organizations that developed methods of analysis in order to present it according to an internationally recognized format as this would facilitate its consideration for endorsement by the CCMAS.

100) Some delegations expressed the view that the method was acceptable from a scientific point of view and supported its endorsement and inclusion in the standard for gluten free foods as this would be a significant progress to address the health problems of coeliac patients.

101) The Committee agreed to inform the Committee on Nutrition and Foods for Dietary Uses of the issues raised concerning the method and to endorse temporarily the Enzyme-Linked Immunoassay R5 Mendez (ELISA) as Type IV, with the understanding that the method would be considered by the next session.

Methods for Additives and Contaminants

102) The Delegation of the Netherlands proposed to amend the type of the two adopted general methods for the determination of heavy metals, using the second method as Type II since microwave digestion provides better results than dry ashing. However the Committee noted that the equipment necessary for this method was not available in all laboratories and the Committee agreed to retain the current types of the methods at this stage. The Committee agreed to clarify that these general methods apply to all foods except fats and oils.

103) The Delegation of the Netherlands informed the Committee that the CEN method for nitrates and nitrites had been published as final CEN methods and the Committee agreed to endorse them as Type III methods. It as noted that the second CEN method was identical to the NMKL method. The Delegation of Morocco pointed out that when spectrometric methods were used for the determination of nitrites interferences with ascorbic acid may occur and it would be preferable to select electrochemical methods that were more selective.

104) The Committee endorsed the other amendments proposed to the methods for additives and contaminants in order to ensure consistency between the methods.

105) The Committee agreed with the proposal of the Delegation of the United Kingdom to initiate the conversion of the methods for trace elements into criteria, for consideration at the next session in the framework of the Agenda Item on Endorsement. The Committee welcomed the proposal of the Observer from NMKL to work in this area with interested delegations and international organisations.

106) The Committee expressed its appreciation to Dr Wood and to the Working Group for their constructive work in order to facilitate the discussions in the Plenary Session and agreed that it would be reconvened prior to the next session. The status of the endorsement of methods of analysis and sampling is presented in Appendix VI.

CRITERIA FOR THE METHODS FOR THE DETECTION AND IDENTIFICATION OF FOODS DERIVED FROM BIOTECHNOLOGY – GENERAL APPROACH AND CRITERIA FOR THE METHODS (Agenda Item 9)[11]

107) The Committee recalled that the last session had agreed that the Delegations of Germany and the United Kingdom in cooperation with a drafting group would prepare a revised document that would include recommendations for quality control measures in laboratories and criteria for methods of analysis.

108) The Delegation of the United Kingdom introduced the document and indicated that it included recommendations on the criteria for methods of analysis and quality control measures that should be introduced in laboratories performing GM analysis, with specific focus the detection of DNA markers based on PCR that were more commonly used.

109) The Delegation of Germany referred to the list of methods developed by the Task Force on Foods Derived from Biotechnology and highlighted the importance of further work on guidelines that would provide guidance to governments to select methods for the detection of foods derived from biotechnology.

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[11] CX/MAS 04/10, CRD 9 (comments of the United States)

110) The Delegation of the United States welcomed the paper that provided a good scientific basis for further discussion and drew the attention of the Committee to its comments in CRD 9. It noted in particular that the document developed criteria mostly for DNA-based methods but that alternative methods based on the detection of protein should also be addressed.

111) The Delegation of Brazil expressed the view that the validation of immunoassay methods should be considered, and that in Annex 1 more information should be included on the description of the method, such as: complete description of the primer, number of cycles, composition of cycles, equipment, amplicon length, type of polymerase and reference material.

112) The Delegation of Japan questioned the application of those criteria contained in the document to the detection of GMOs although they are applicable to chemical analysis.

113) The Delegation of Norway proposed to amend the section on the modular approach to reflect that it should not be used "unless independence between the modules can be documented", since it should not be systematically avoided.

114) The Delegation of Cuba drew the attention of the Committee to the issues related to consumer protection, that might need to be addressed by the Task Force in the future and in particular the level of transgenicity of the material.

115) The Committee discussed whether new work should be initiated in the Step Procedure in order to circulate for comments as soon as possible the document in Appendix I: *Guidelines for the Validation and Quality Control Requirements for GMO Analyses*.

116) Some delegations stressed the need to proceed rapidly as governments needed guidance on this very important and complex issue. Other delegations indicated that they had been part of the original Working Group but there had not been enough time to provide detailed comments and that it would be preferable to consider the text carefully before initiating the elaboration of specific guidelines.

117) The Committee agreed that the document would be revised by the Delegations of the United Kingdom and Germany with the assistance of a Drafting Group[12] for consideration at the next session, with a view to the elaboration of Guidelines.

METHODS OF ANALYSIS FOR THE DETERMINATION OF DIOXINS AND PCBS (Agenda Item 10)[13]

118) The Delegation of Germany expressed its deep regret that due to the lack of input it had not been possible to prepare a list of methods for determination of dioxins and PCBs for consideration at this session. The Delegation drew the attention of the Committee to the fact that the WHO and EC had started a joint project related to rapid assays for dioxins and related compounds (see CX/MAS 04/INF1) which would provide an additional input in preparing a more comprehensive list of methods for determination of the above compounds.

119) The Committee agreed that a Circular Letter would be prepared to seek information on the current methods used for determination of dioxins and related compounds. This information should be forwarded to Germany who would prepare a paper for consideration at the next session of the Committee.

REPORT OF AN INTER-AGENCY MEETING ON METHODS OF ANALYSIS AND SAMPLING (Agenda Item 11)[14]

120) The Chairman of the Inter-Agency Meeting (IAM) Dr Roger Wood introduced the draft report of the 16th IAM and informed the Committee that CRD 2 was being presented mainly for information purposes and highlighted the following important issues discussed at the IAM.

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12 Argentina, Australia, Brazil, Canada, Egypt, European Community, France, Iran, Ireland, Italy, Japan, Malaysia, Netherlands, Norway, Philippines, United States, AOAC, AOCS, BIO, CROPLIFE International, EUROPABIO and ISO

13 CX/MAS 04/INF 1 (Draft report on the Symposium on Rapid Assays for Dioxins and related Compounds)

14 CRD 2 (Report of the 16th Meeting of international organizations working in the field of methods of analysis and sampling (Inter-Agency Meeting)

121) It was indicated that the criteria approach had been adopted by the Commission and that the validation data would have to be made available to customers to ensure a successful application of this approach.

122) Dr Wood drew the attention of the Committee to the problem related to the application by laboratories of proficiency testing and noted that high level laboratories could provide reference values for particular difficult analyses.

123) The Committee was informed about the progress of the electronic compendium of analytical methods and that validated methods of analysis should be made generally available.

124) As regards the question of incorporation of changes to methods/method corrections in the Codex Alimentarius, it was indicated that the current system was retained.

125) The Committee was informed that the paper on harmonization of analytical terminology would be updated and available from the website.

126) Finally Dr Wood informed the Committee about the discussions on the Terms of Reference of the IAM and future changes in the Secretariat.

127) The Committee expressed its appreciation to the IAM and Dr Wood for their constructive work and contribution to the work of the Committee and noted that the final report would be made available from the website of AOAC.

OTHER BUSINESS AND FUTURE WORK (Agenda Item 12)

The Use of Analytical Results: Sampling Plans, Relationship Between the Analytical Results, the Measurement Uncertainty, Recovery Factors and Provisions in Codex Standards

128) The Committee recalled that it had agreed to consider the Guidelines redrafted by the *Ad Hoc* Working Group under Agenda Item 12 "Other Business and Future Work" (see also paras 6) to 13).

129) The Chairperson of the *Ad Hoc* Working Group presented the document and indicated that the text was substantively revised and it should give simple instructions to Commodity Committees regarding sampling, relationship between the analytical results, the measurement uncertainty, recovery factors and provisions in the Codex standards.

130) The Committee amended the title to reflect the view that the use of analytical results related not to sampling as such but rather to sampling plans.

131) The Delegation of New Zealand was of the view that there was a contradiction between the first and last paragraphs of the section on Issues Involved and this required further rewording for clarification purposes. It proposed several substantial amendments as follows. The section on Measurement Uncertainty was not sufficiently general, as this was not the only way to proceed, and should be addressed in a separate paper. The sampling plan should specify whether the specification applied to the average in a lot or the proportion of non-conforming; significant figures should not be addressed in the document as this question relates to reporting not to the use in conformity assessment.

132) The Committee amended the first paragraph in the Recommendations section to clarify that when Commodity Committees discuss and agree on a specification, the concerned analytical methods should also be stated.

133) Different views were expressed regarding the section on Recovery. The Delegation of Ireland drew the attention of the Committee to the fact that recovery was relevant to organic analysis especially when low levels were analysed and proposed to amend the sentence so that the analytical results are to be reported on recovery "where relevant and appropriate".

134) Some delegations proposed to delete this section while other delegations were of the view that the two first sentences from the earlier version of the document better reflected recommendations regarding recovery. The Committee agreed to amend this section as proposed by the Delegation of Ireland and retained it in square brackets for further discussion.

135) It was proposed to forward the document to the Committee on General Principles for their endorsement and subsequent adoption by the Commission and inclusion in the Procedural Manual as guidance to the Codex Commodity Committees. However the Committee noted that although the document was substantively improved, several issues remained to be addressed and there was a need for further consideration therefore decided to request comments on the current version and consider it at the next session of the Committee (see Appendix VII). It also agreed that the advice of Commodity Committee would be sought on this document.

FUTURE WORK

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

- Proposed Draft Guidelines for Evaluating Acceptable Methods of Analysis
- Consideration of the Fitness-For Purpose Approach to Evaluating Methods of Analysis
- The Use of Analytical Results: Sampling Plans, Relationship between the Analytical Results, the Measurement Uncertainty, Recovery Factors and Provisions in Codex Standards
- Proposed Draft Guidelines for Settling Disputes Over Analytical (Tests) Results
- Review of Analytical Terminology for Codex Use in the Procedural Manual
- Endorsement of Methods of Analysis Provisions in Codex Standards, including Conversion of Methods for Trace Elements into Criteria
- Criteria for the Methods for the Detection and Identification of Foods derived from Biotechnology
- Methods of Analysis for Determination of Dioxins and Related Compounds
- Report of an Inter-Agency Meeting on Methods of Analysis

DATE AND PLACE OF THE NEXT SESSION (Agenda Item 13)

137) The Committee was informed that the 26th Session of the Committee would be held in Budapest from 4 to 8 April 2005. The exact venue would be determined by the host country and the Codex Secretariat.

SUMMARY STATUS OF WORK

Subject Matter	**Step**	**Action by**	**Document Reference in ALINORM 04/27/23**
Proposed amendments to the Procedural Manual: - *General Criteria for the Selection of Single-Laboratory Validated Methods of Analysis* - *Analytical Terminology for Codex Use*		CCGP Governments 27th CAC	paras. 18 and 72 Appendix II
Draft General Guidelines on Sampling	8	Governments 27th CAC	para. 25 Appendix III
Draft Guidelines on Measurement Uncertainty	8	Governments 27th CAC	para. 37 Appendix IV
Proposed Draft Guidelines for Evaluating Acceptable Methods of Analysis	5	Governments 27th CAC 26th CCMAS	para. 54 Appendix V
Endorsement of methods of analysis, including general methods		Governments 27th CAC	paras. 77-106 Appendix VI
Fitness-for-purpose Approach (for inclusion in the *Proposed Draft Guidelines for Evaluating Acceptable Methods of Analysis*)	2/3	United Kingdom Governments 26th CCMAS	para. 65
Proposed Draft Guidelines for Settling Disputes on Analytical (Test) Results	2/3	France/Governments 26th CCMAS	para. 58
Use of Analytical Results	(*)	Commodity Committees Governments 26th CCMAS	para. 135 Appendix VII
Further Review of *Analytical Terminology for Codex Use* (Procedural Manual)	(*)	Governments 26th CCMAS	para. 76
Criteria for methods of analysis for foods derived from biotechnology		United Kingdom/ Germany Governments 26th CMAS	para. 117
Methods of analysis for dioxins and PCBs		Germany/Governments 26th CCMAS	para. 119

(*) For inclusion in the Procedural Manual

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ALINORM 04/27/23
APPENDIX II

PROPOSED AMENDMENTS TO THE PROCEDURAL MANUAL

1. GENERAL CRITERIA FOR THE SELECTION OF SINGLE-LABORATORY VALIDATED METHODS OF ANALYSIS (TO BE INCLUDED AFTER THE GENERAL CRITERIA)

Inter-laboratory validated methods are not always available or applicable, especially in the case of multi-analyte/multi substrate methods and new analytes. The criteria to be used to select a method are included in the General Criteria for the Selection of Methods of Analysis. In addition the single-laboratory validated methods must fulfill the following criteria:

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

ii. the use of the method is embedded in a quality system in compliance with the ISO/IEC 17025: 1999 Standard or the OECD Principles of Good Laboratory Practice;

The method should be complemented with information on accuracy demonstrated for instance with:

- regular participation in proficiency schemes, where available;
- calibration using certified reference materials, where applicable;
- recovery studies performed at the expected concentration of the analytes;
- verification of result with other validated method where available

2. Guidelines for the Inclusion of Specific Provisions in Codex Standards and Related Texts

Principles for the Establishment of Codex Methods of Analysis

AMENDMENTS TO ANALYTICAL TERMINOLOGY FOR CODEX USE

Specificity: deleted

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

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.

Accuracy (as a concept) and Accuracy (as a statistic) to be replaced with the following definition:

Accuracy: The closeness of agreement between a test 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.

Trueness**:** The closeness of agreement between the average value obtained from a series of test results and an accepted reference value.

Notes:

1 The measure of trueness is usually expressed in terms of bias.

2 Trueness has been referred to as “accuracy of the mean”. This usage is not recommended.

TERMS TO BE USED IN THE CRITERIA APPROACH

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

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.

PROPOSED DRAFT GENERAL GUIDELINES ON SAMPLING
(At Step 8 of the Procedure)

TABLE OF CONTENTS

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]

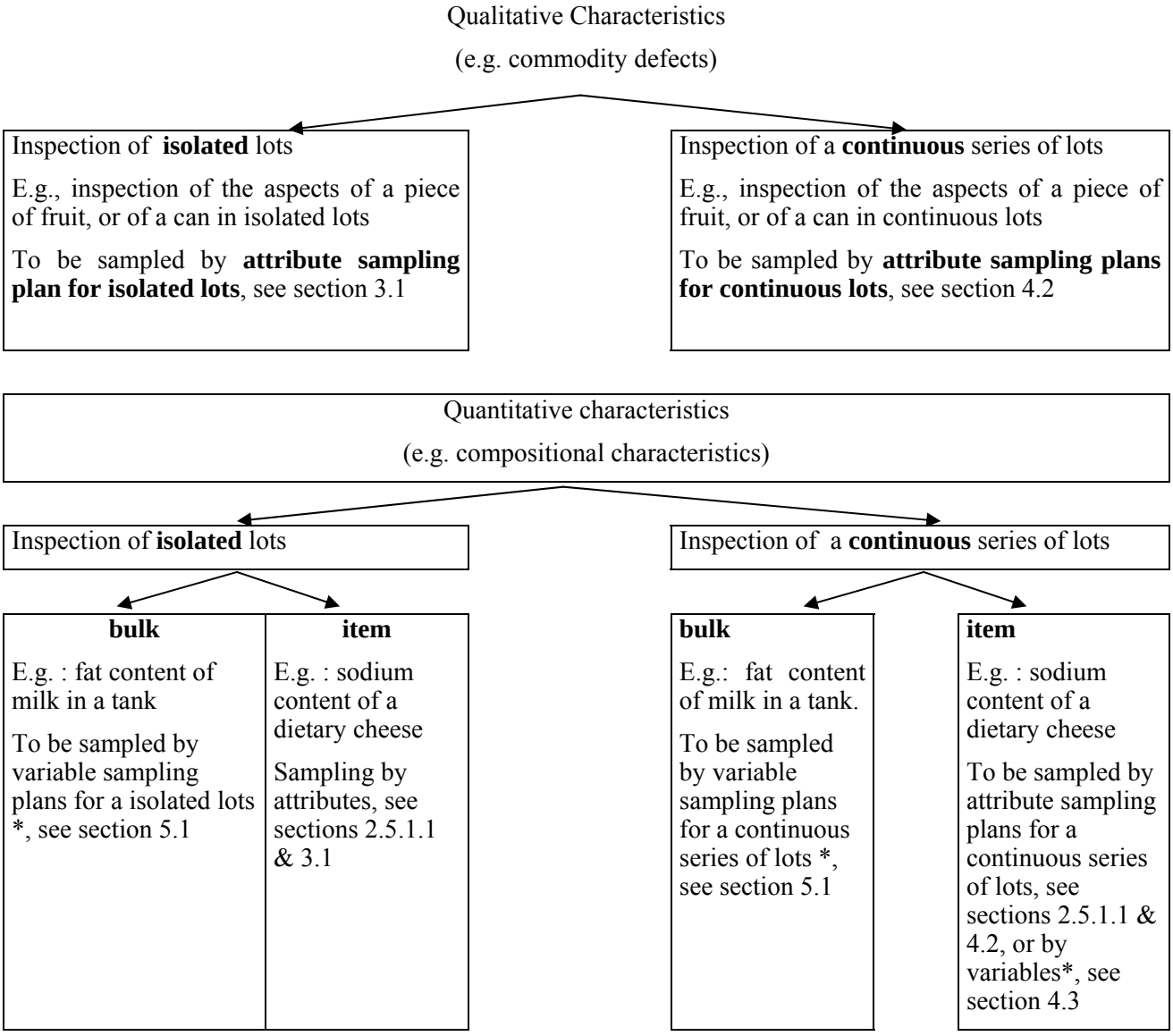
1) **Existence (or not) of international reference documents** on sampling of the considered products
2) **Nature of the control**
   - Characteristic applicable to each individual item of the lot
   - Characteristic applicable to the whole lot (statistical approach)
3) **Nature of the characteristic 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)
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).

[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.

- 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.

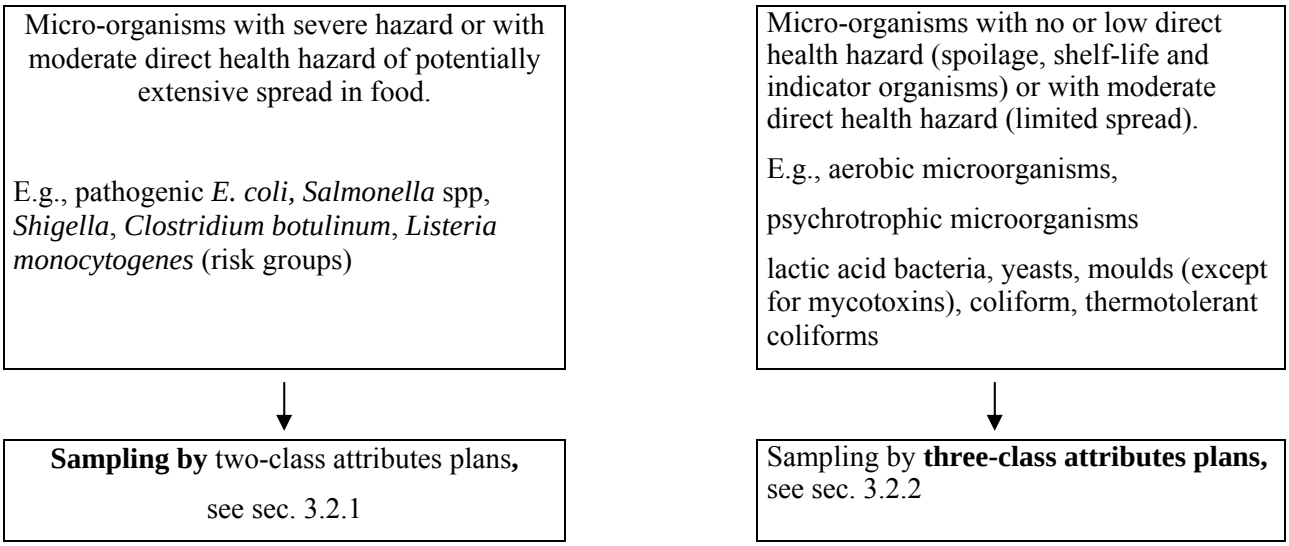
FLOW-CHART FOR CHEMICAL AND PHYSICAL CHARACTERISTICS



* normal distribution is assumed

[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 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 (see 2.4), 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 bulk material	**Lots consisting of individual[4] items**		
	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 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)
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)

[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.

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 5725-1:1994 (E): Application of statistics – Accuracy (trueness and precision) of measurement methods and results – Part 1: General principles and definitions

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

2.1 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

[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.

acceptable quality and the limiting quality level (section 2.2.14). These notions are essential for risk assessment prior to selecting a plan;

- 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.

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.

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 (see 3.2.3 for some examples).

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 (such as health-related properties, see Table 2);
- Class B: Those nonconformities considered to be less important than the Class A nonconformities (such as commodity defects or compositional characteristics, see Table 2).

This classification should be determined by the Codex Commodity Committees.

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 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.

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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).

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 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.

D = CR - PR

Discrimitation ratio (DR)

The discrimination ratio (DR) is the ratio between the consumers' risk (CR) and the producers'risk (PR). It is generally given by the ratio between P_{10} and P_{95}.

$$DR = \frac{P_{10}}{P_{95}}$$

This ratio enables to appreciate also the efficiency of a sampling plan. A ratio below 35[9] characterises a sampling plan with a particularly low efficiency.

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

---

[9] The DR of an attribute sampling plan (n=2, c=0) is 27, the one of an attribute sampling plan (n=3, c=0) is 32, the one of an attribute sampling plan (n=5, c=0) is 36.

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.

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. However, the designers of the plans in the ISO and other reference documents have deliberately introduced a relationship to reduce the risk of making an incorrect decision for larger lots. The ratio f = n/N influences the sampling error only 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 be 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.

Refer to the tables of ISO 2859 and ISO 3951 for correspondence between sample size and lot size.

2.3 SAMPLING PROCEDURES

2.3.1 General

Sampling procedures should be performed in accordance with appropriate ISO Standards related to the commodity of concern (for example ISO 707 for sampling of milk and milk products).

2.3.2 Employment of Sampling Officers

Sampling should be performed by persons trained in the techniques of sample collection by the importing country.

2.3.3 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.

Representative sampling

The representative sampling is a procedure used for drawing or forming a representative sample[10].

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[11], 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,…

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

[11] 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.

2.3.5 Preparation of samples

2.3.5.1 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 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).

2.3.5.2 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.

2.3.5.3 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.

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.

2.3.7 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.

The total standard deviation σ is given by the formula:

$$\sigma = \sqrt{\sigma_s^2 + \sigma_m^2}$$

where σ_s is the sampling standard-deviation, σ_m the measurement standard-deviation

- First case (the most frequent one) : the analytical error is negligible compared to the sampling error, i.e the analytical error is at most equal to one third of the sampling error

  In this case, $\sigma_m \leq \sigma_s/3$, and $\sigma \leq \sqrt{\sigma_s^2 (1 + 1/9)} = 1{,}05 \times \sigma_s$

  The standard deviation for the observed results will be at most 5 % larger than the sampling standard deviation taking into account the analytical error.

– Second case: the analytical error is larger than one third of the sampling error

  This case is not covered by these Guidelines.

2.5 TYPES OF SINGLE SAMPLING PLANS

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

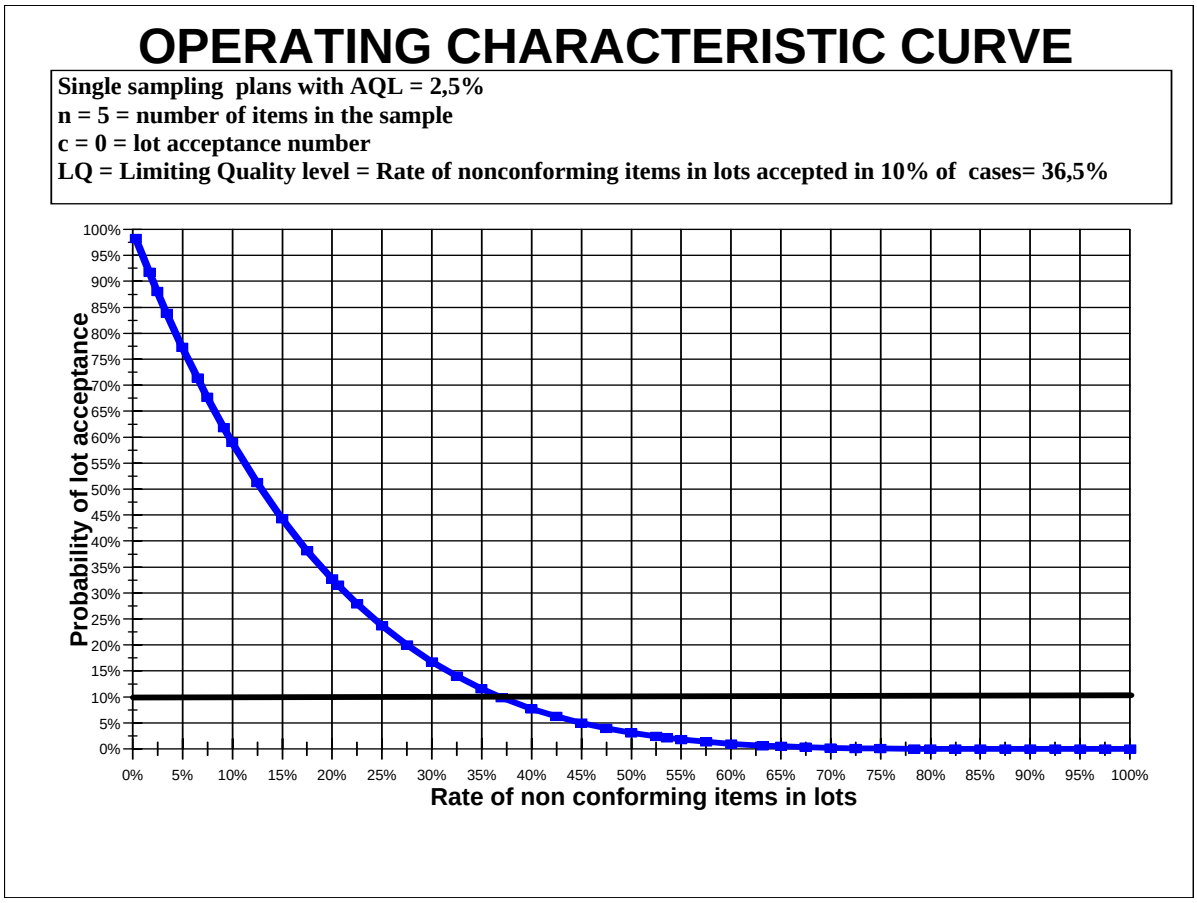
2.5.1.1 Principles of inspection by attributes of percent non-conforming items

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.

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 nonconforming

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 $\bar{x}$ 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 %.

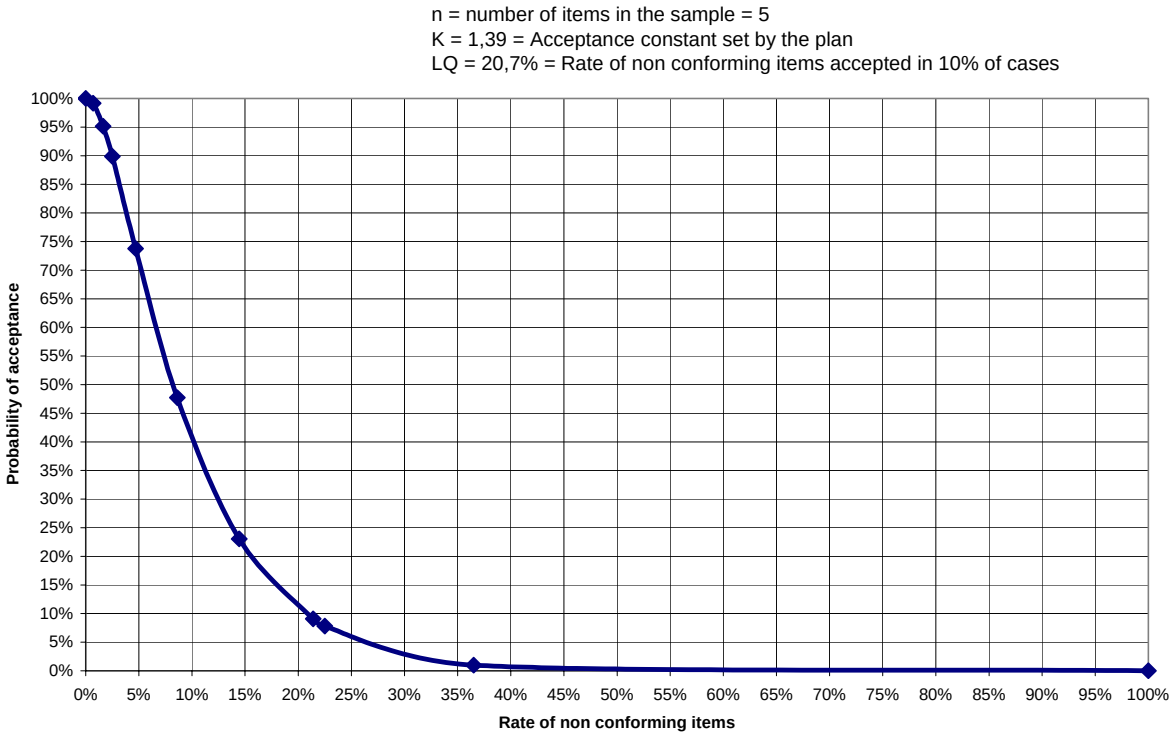
- σ = 3,5 mg, the known standard deviation according to experimental data on an extended period of production, made available to the inspectors by the professionals.

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 - Kσ and the lot is rejected.
- The operating characteristic curve of the plan by variables is given in the figure 2.

Figure 2: OC curve, single sampling plan by variable, known standard deviation



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[12] :

- 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).

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

[12] 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.

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: Comparison of OC curves of variable sampling plans : s-method and σ-method, same AQL (2,5 %) and same sample sample size (5 items)*

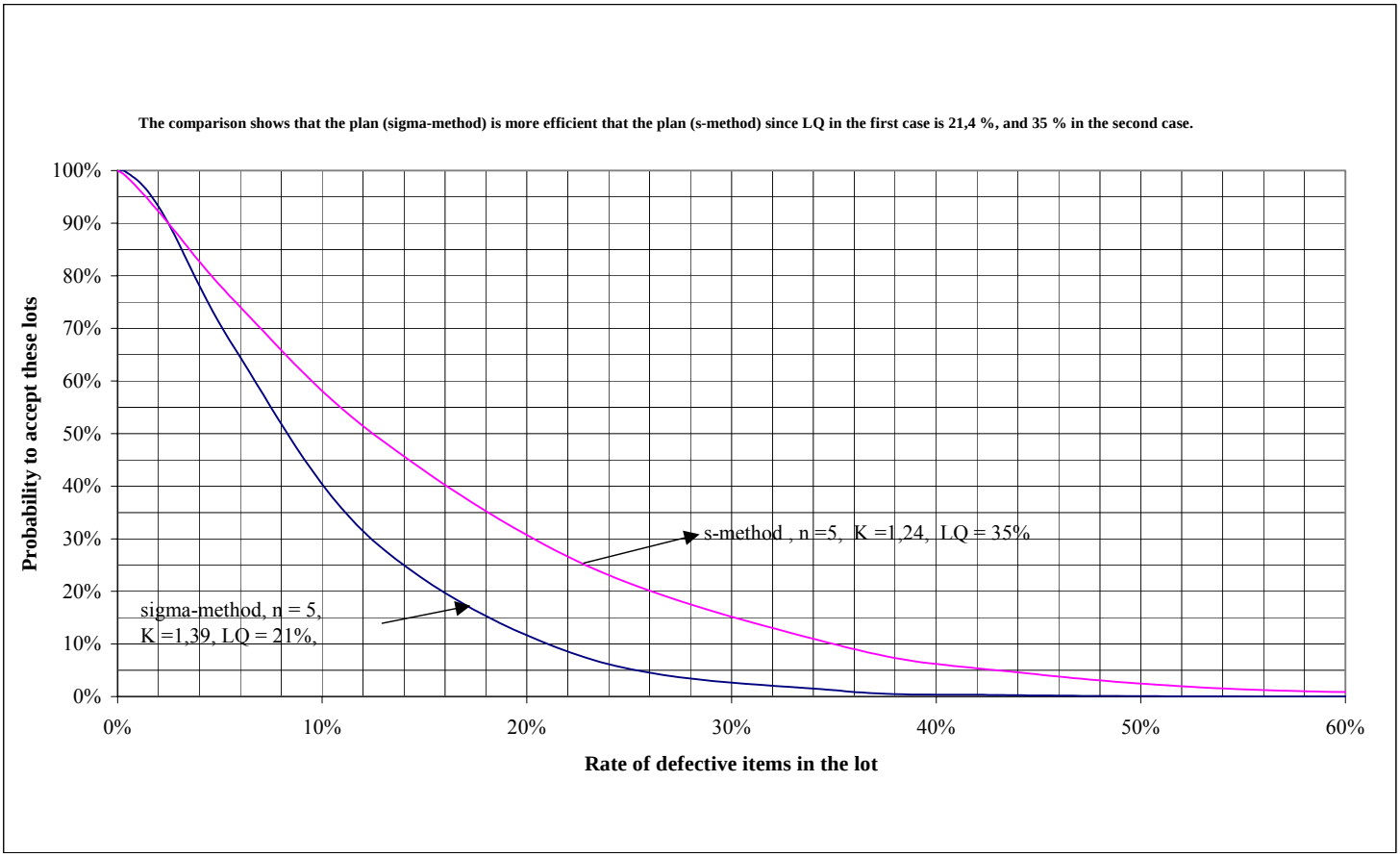


Table 5: Probability of lot acceptance by defective rates and sampling method (s-method, σ-method)

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: **Comparison of OC curves of a variable and an attribute sampling plans**

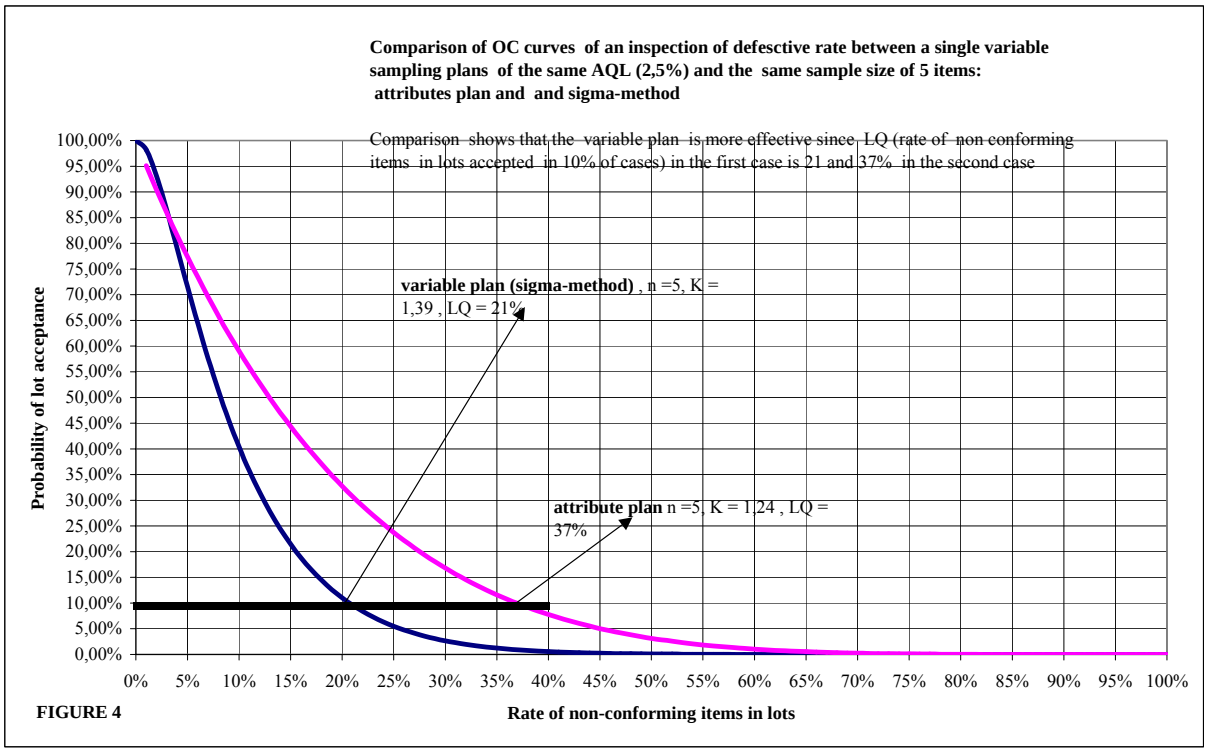
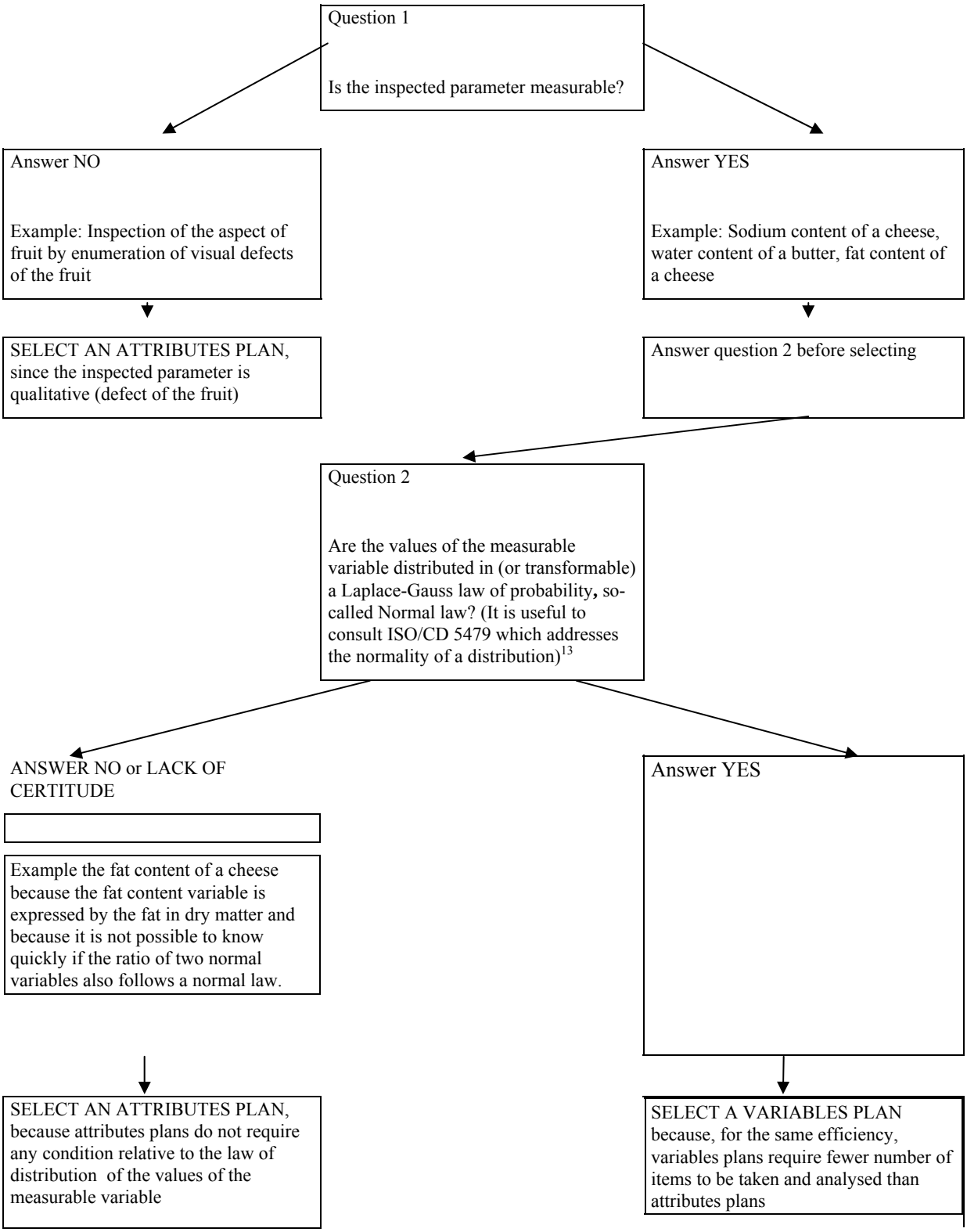


FIGURE 4

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:



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

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[14].

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:

Table 7: Comparison of sample sizes for attribute and variable sampling plans (normal inspection level) by Sample Size and Code Letter

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.

[14] 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.

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[15] : 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:

---

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

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

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[16]

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:

(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

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

⬇

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.

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

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

$s = \sqrt{\dfrac{\sum_{i=1}^{n} \left(x_i - \bar{x}\right)^2}{n-1}}$ 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[17].

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

Decision Rules

The lot is accepted if:

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

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

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

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)[18] 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

[18] 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.

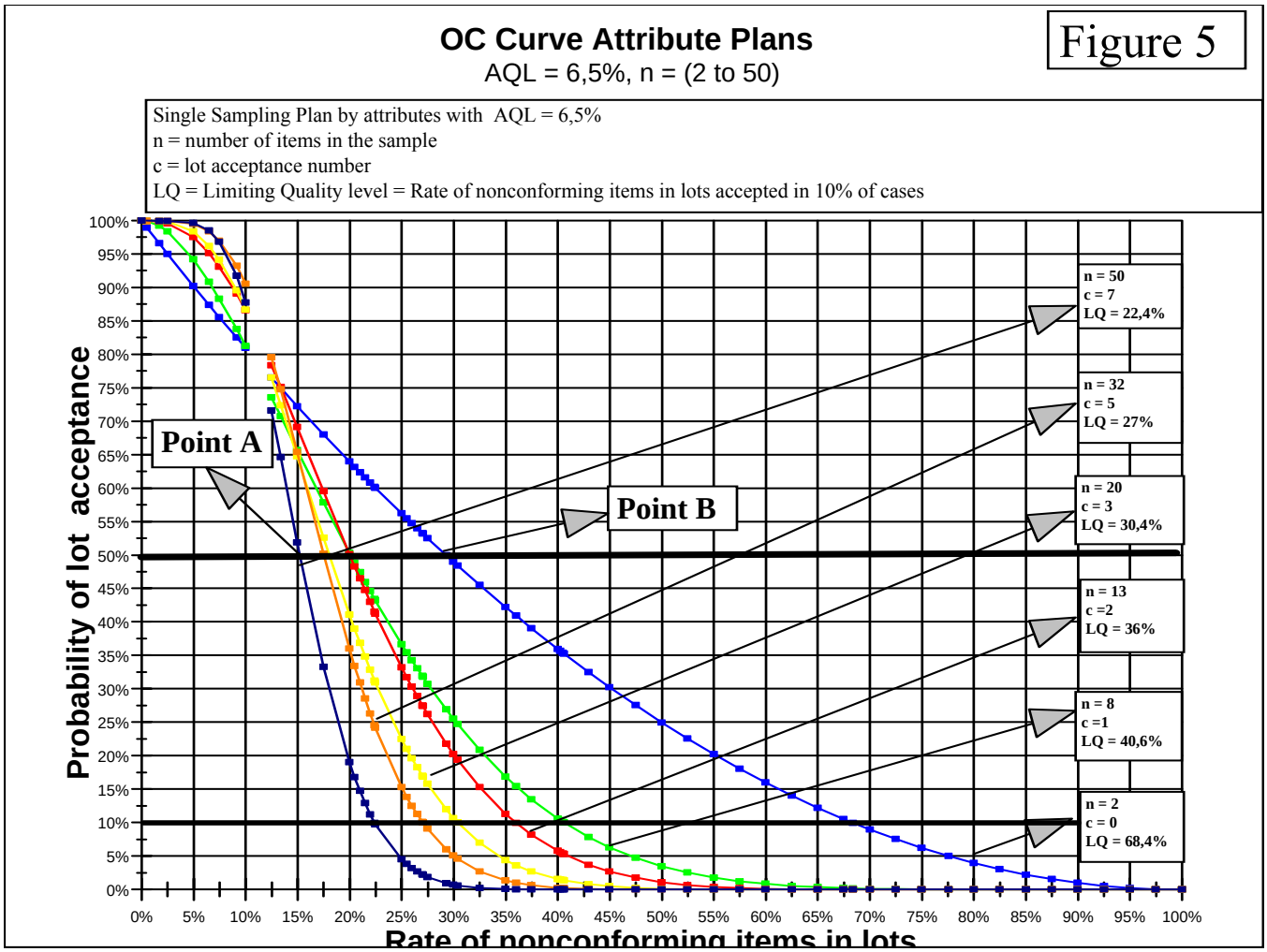
The curve of Figure 5, which contains the point A, corresponds to a lot inspected with a 50-item sample. The lot is accepted at inspection if there are less than 7 defective items in the sample. The abscissa of the point A (15 %) corresponds to a lot containing 15 % of defective items; its ordinate (50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

Rate of nonconforming items in lots



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[19], tightened, reduced)

⬇

Set the AQL

⬇

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

⬇

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

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).

Table 10. Attribute Sampling Plan

		Inspection level		
Lot size (Number of items)		**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

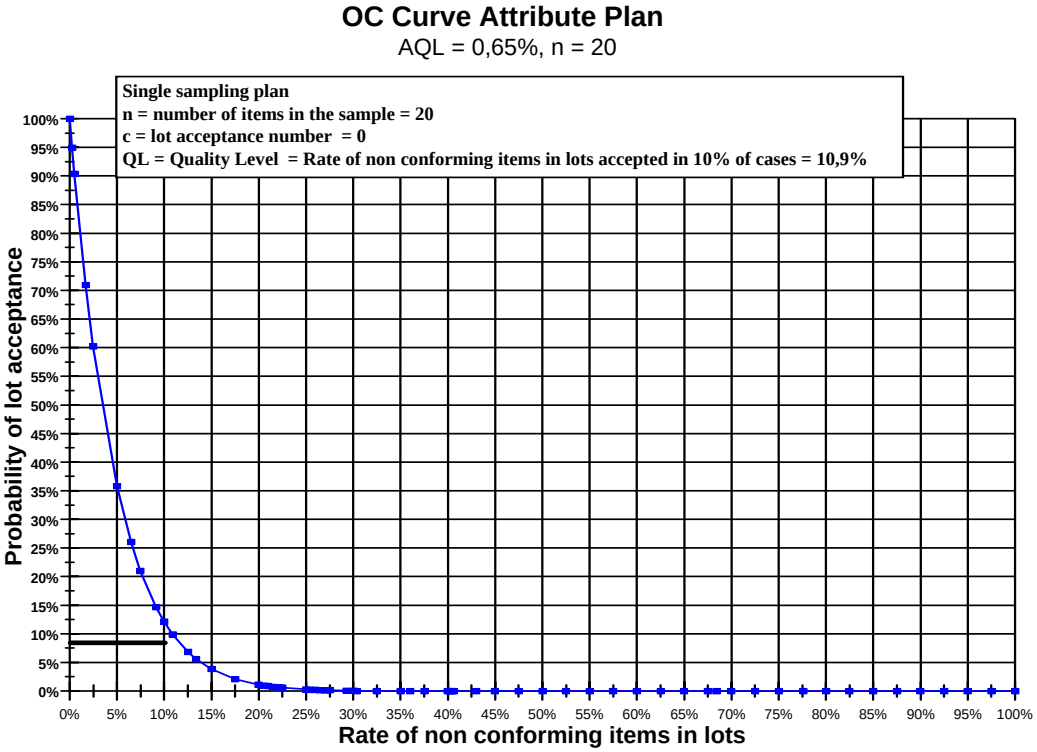
	c at AQL = 6,5	0	2	2
91 - 150	n	**3**	**20**	**32**
	c at AQL = 0,65	0	0	0
	c at AQL = 2,5	0	1	1
	c at AQL = 6,5	0	3	3
151 - 280	n	**5**	**32**	**50**
	c at AQL = 0,65	0	0	1
	c at AQL = 2,5	0	2	2
	c at AQL = 6,5	1	5	5
281 - 500	n	**8**	**50**	**80**
	c at AQL = 0,65	0	1	1
	c at AQL = 2,5	0	3	3
	c at 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
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 %

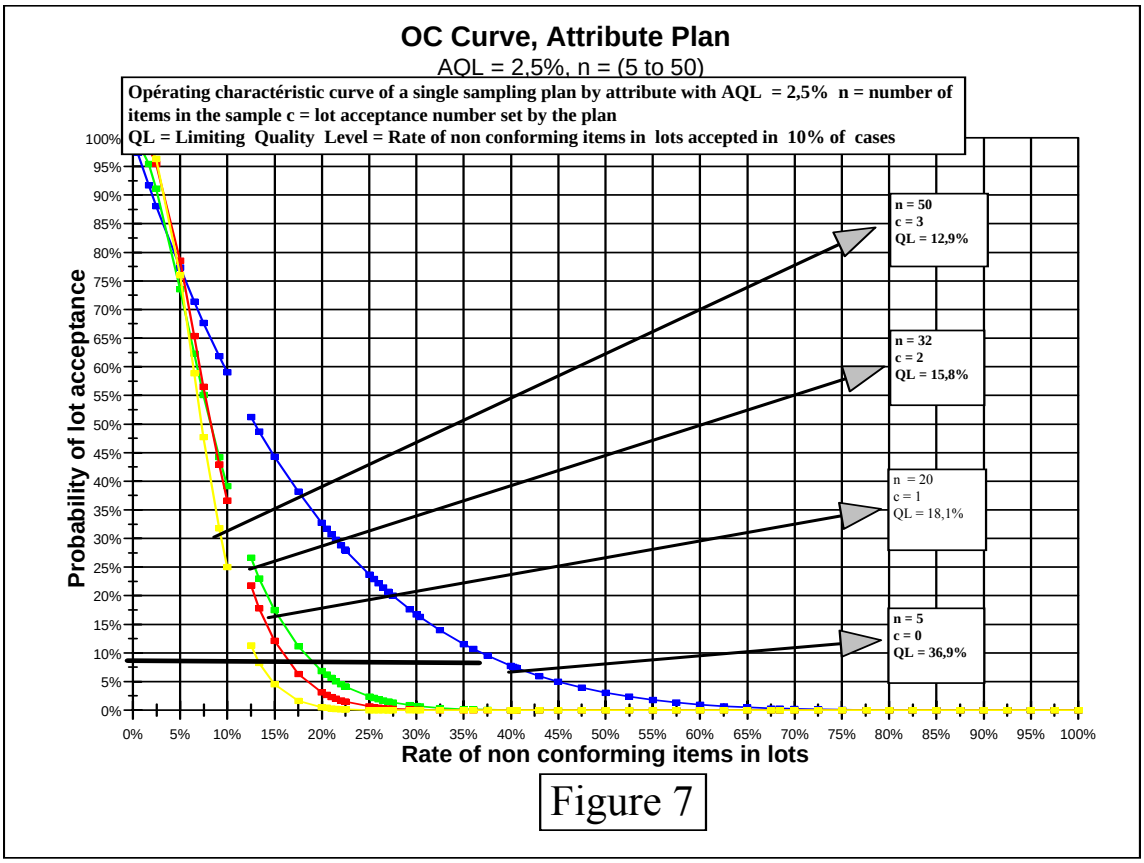


4.2.2.2 Plans with AQL = 2,5% (see Table 12 and figure 7)

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%
[2]100%	0%	0%	0%	0%

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



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} [20]= 2,53% P_{50} [21]=29,3% P_{10} [22] =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%

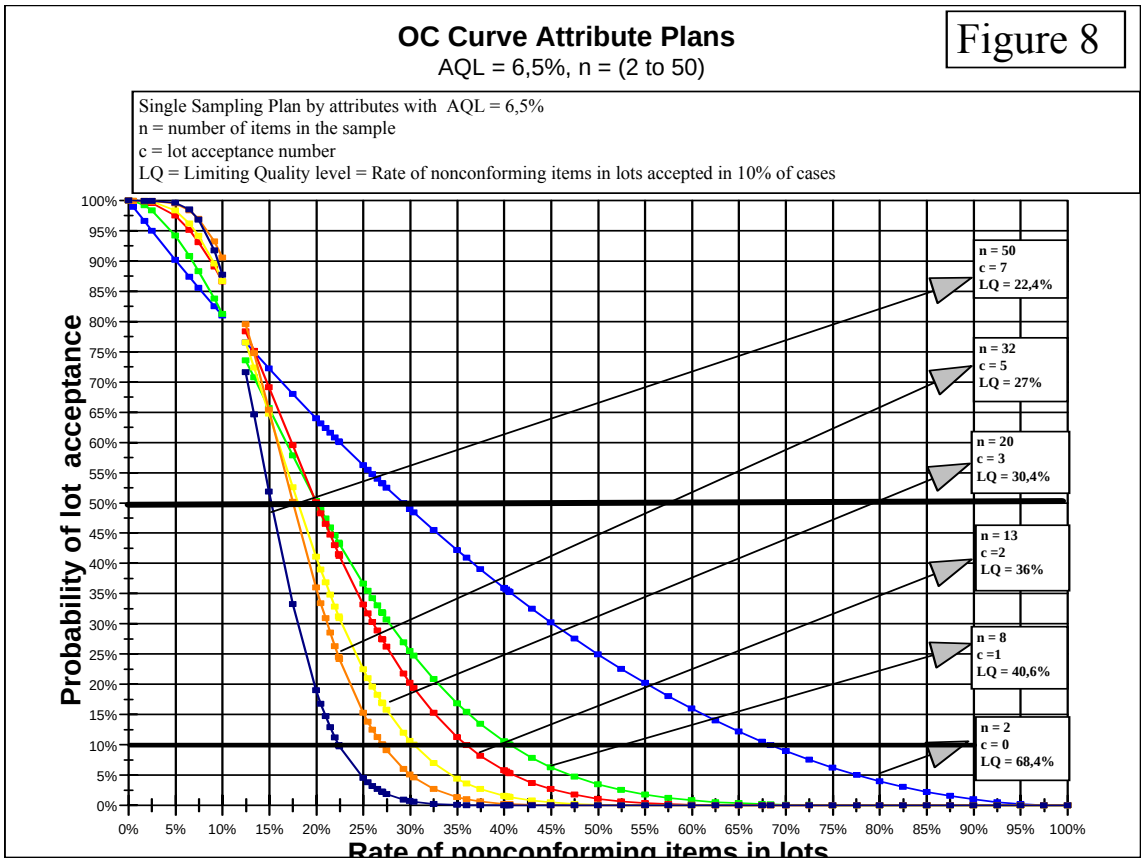
[20] P_{95} = Rate of non-conforming items in lots accepted in 95% of cases

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

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

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 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 professionals.

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 using the following approximation:

$$u_{PA} = \frac{\sqrt{n} \times (u_{1-p} - K)}{\sqrt{1 + K^2/2}}$$

where:

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

P_A is the probability of acceptance of a lot containing a defective rate of p,

K is the acceptability constant,

u_{1-p} is the fractile of order 1-p of the standardized normal law,

n is the sample size.

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 (Number of items)	**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
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 k at 0,65 k at 2,5 k at 6,5	**35** 1,89 1,39 0,969	**200** 2,18 1,70 1,29	**200** 2,33 1,89 1,51
500 001 and over	n k at 0,65 k at 2,5 k at 6,5	**50** 1,93 1,42 1,00	**200** 2,18 1,70 1,29	**200** 2,33 1,89 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} [23]= 0,28% P_{50} [24]= 6,34% P_{10} [25] = 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%
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%

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

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

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

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} [26]= 0,49% P_{50} [27]= 2,69% P_{10} [28] = 7,46%	Letter-codeIE, AQL = 0,65%, n= 25, K =1,96 P_{95} = 0,56% P_{50} = 2,53% P_{10} = 6,46%	Letter-code J, AQL = 0,65%, n= 10, K =1,84 P_{95} = 0,36% P_{50} = 3,77% P_{10} = 13,2%	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%

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

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

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

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

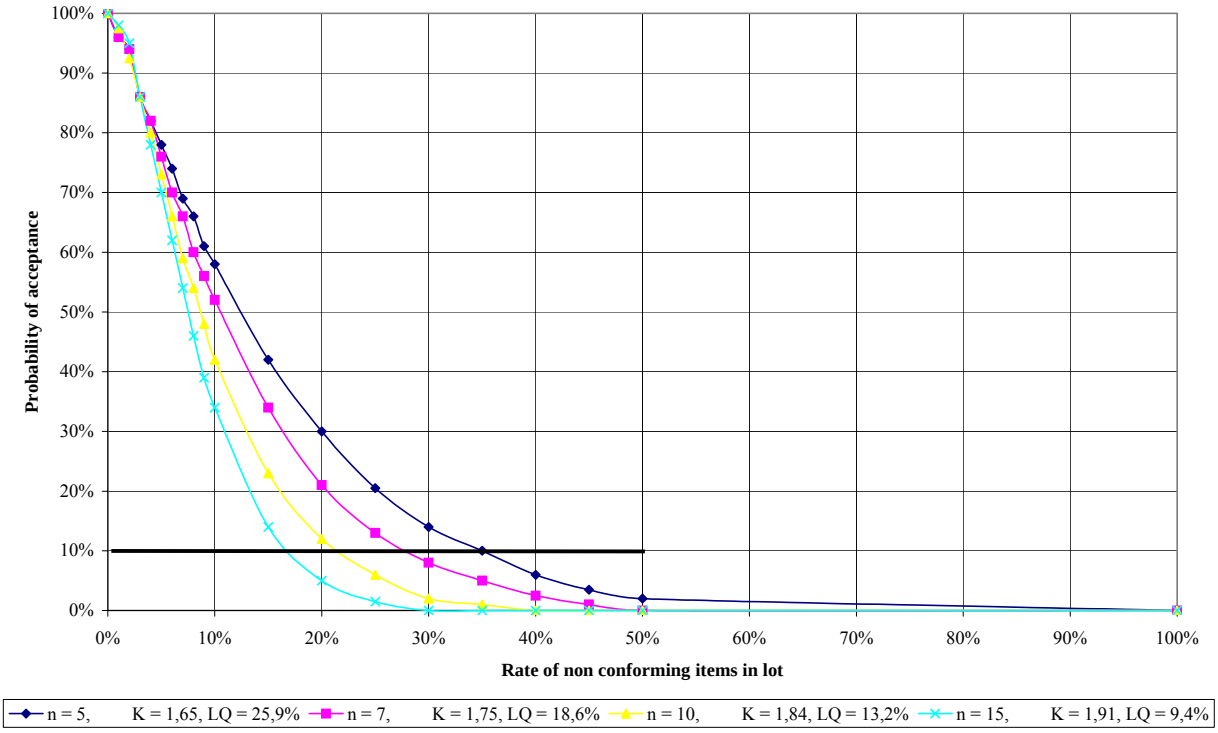
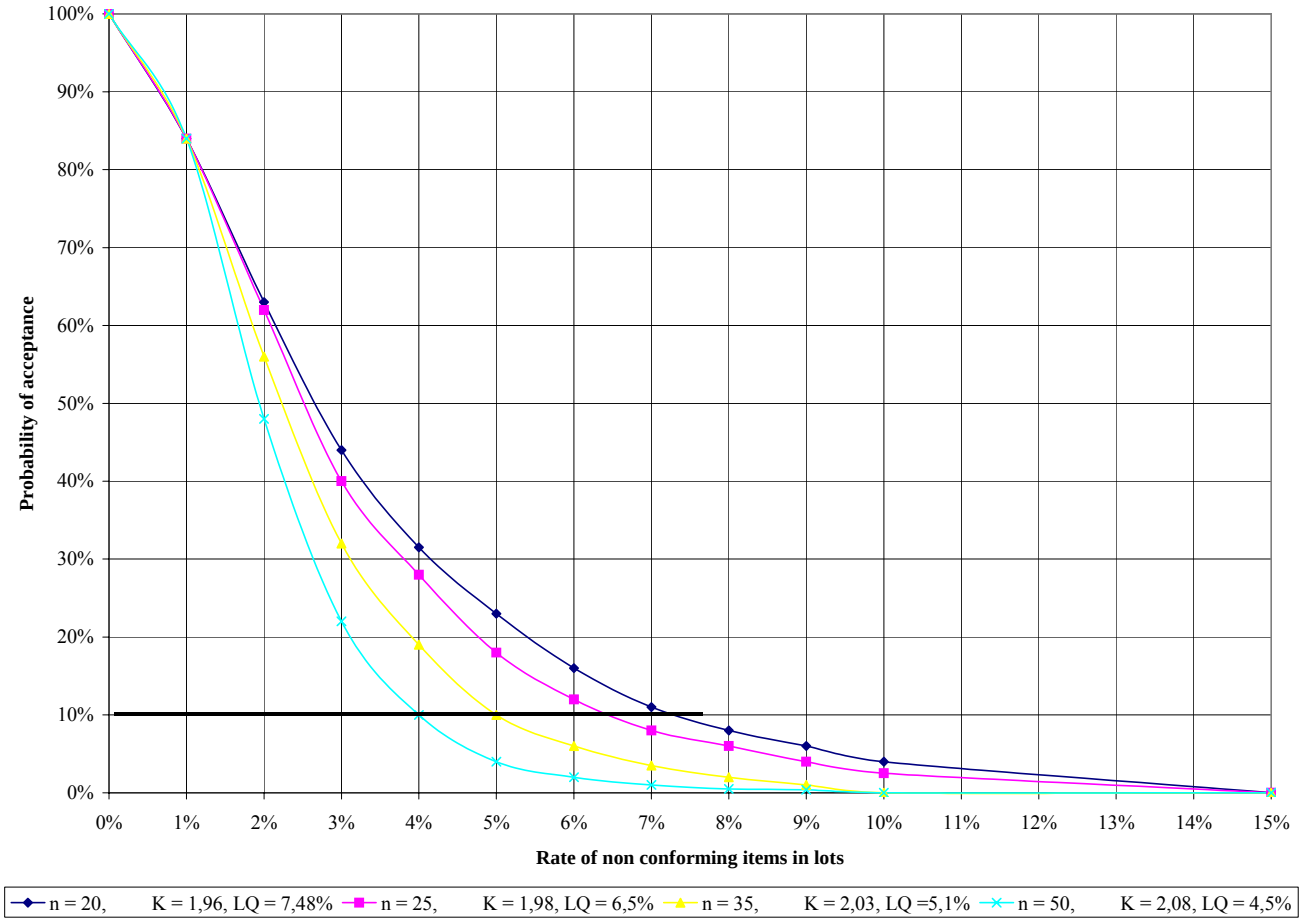


Figure 10: OC curve, variable sampling plan, s-method, AQL = 0,65 %, n = 20 to 50



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, s-method, AQL = 2,5 %, n = 5 to 15

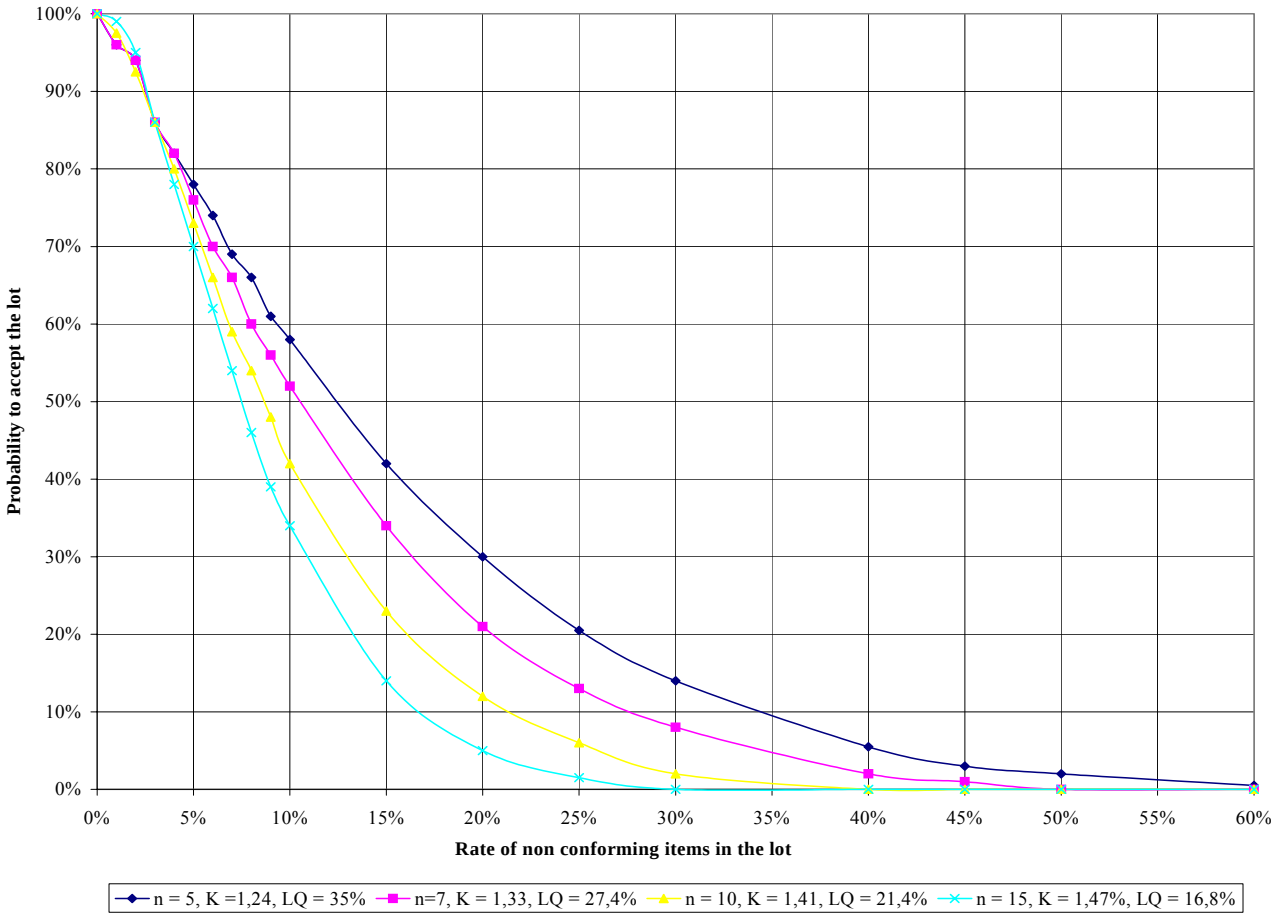
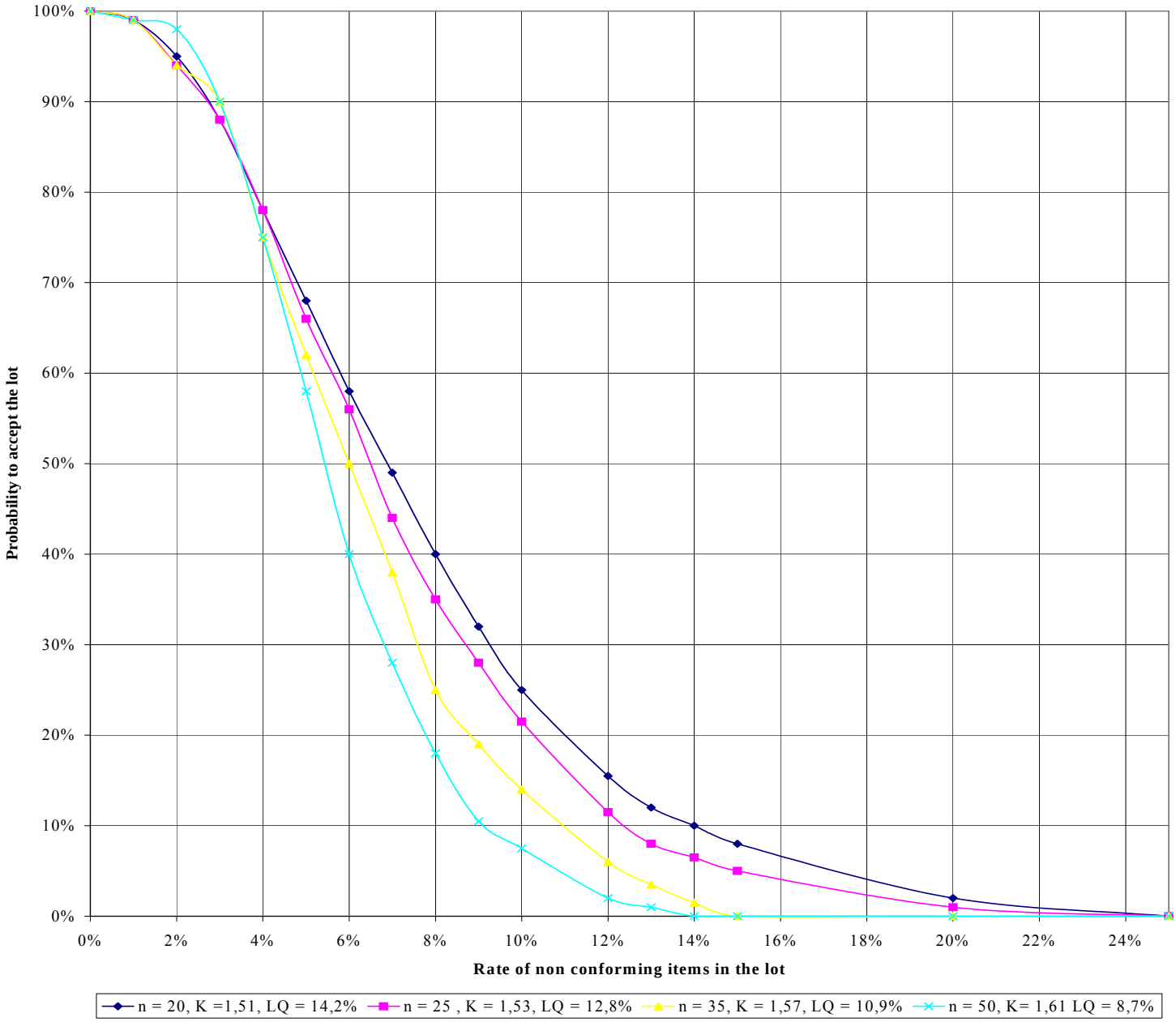


Figure 12: OC curve, variable sampling plan, s-method, AQL = 2,5 %, n = 20 to 50



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 of accepting a 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 Procedure 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 (Number of items)	**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

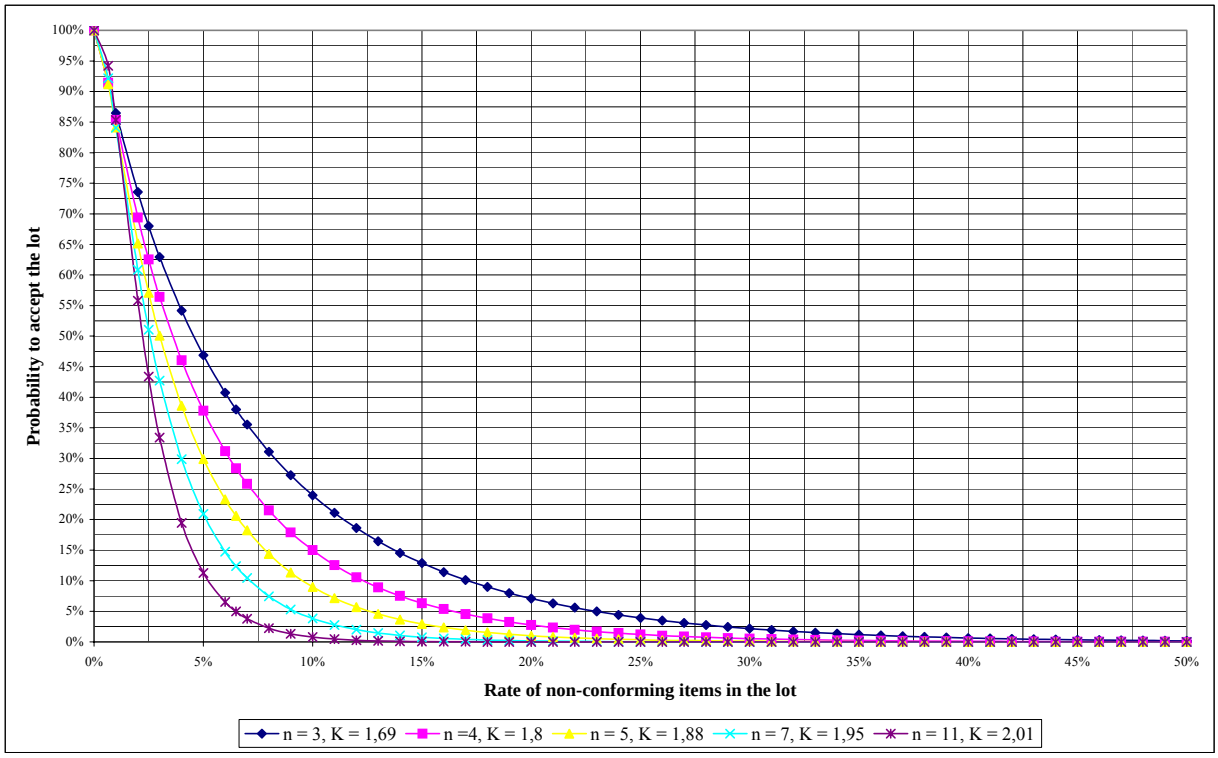
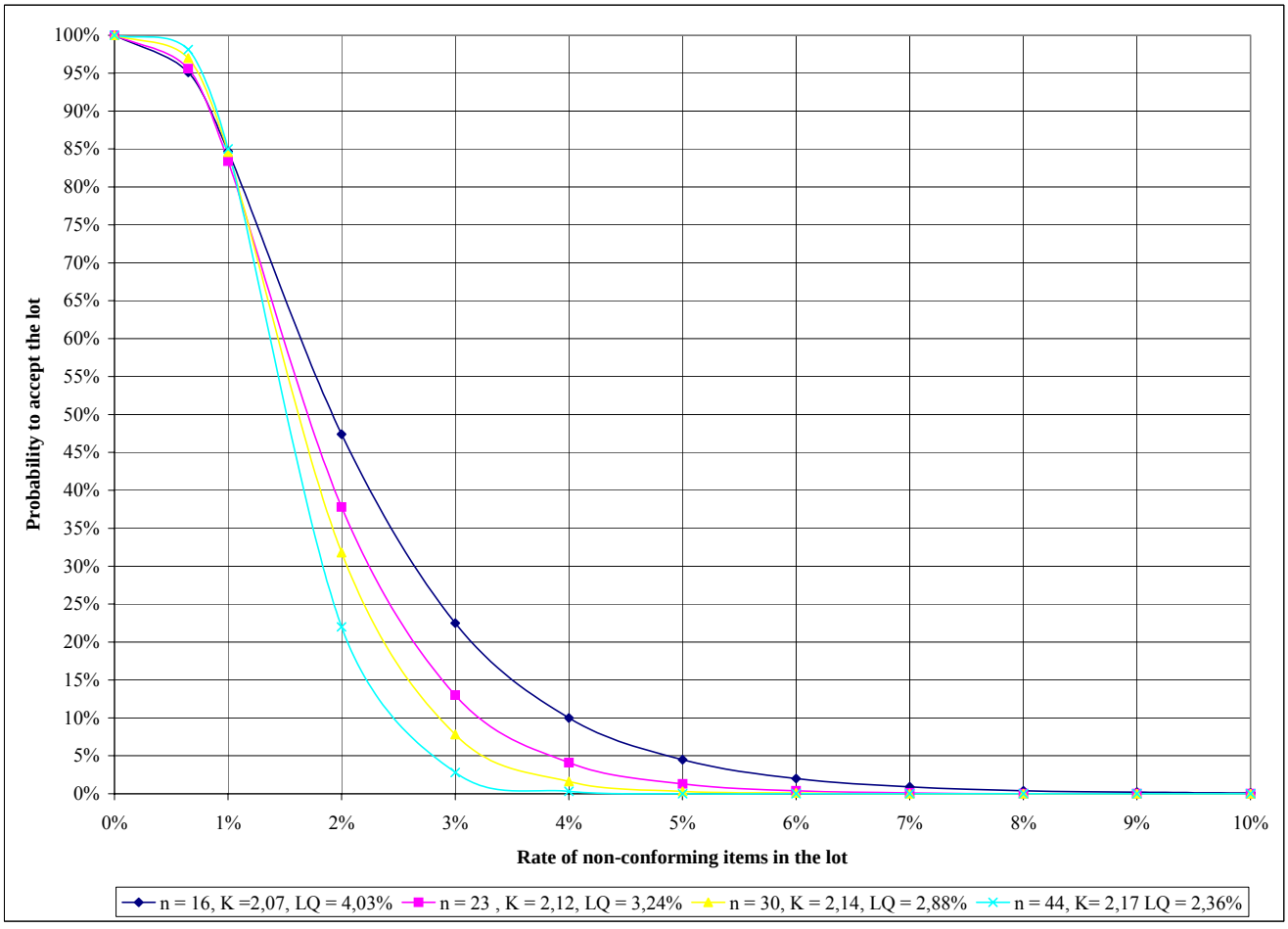


Figure 14: OC curve, variable sampling plan, σ-method, AQL = 0,65 %, n = 16 to 44



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

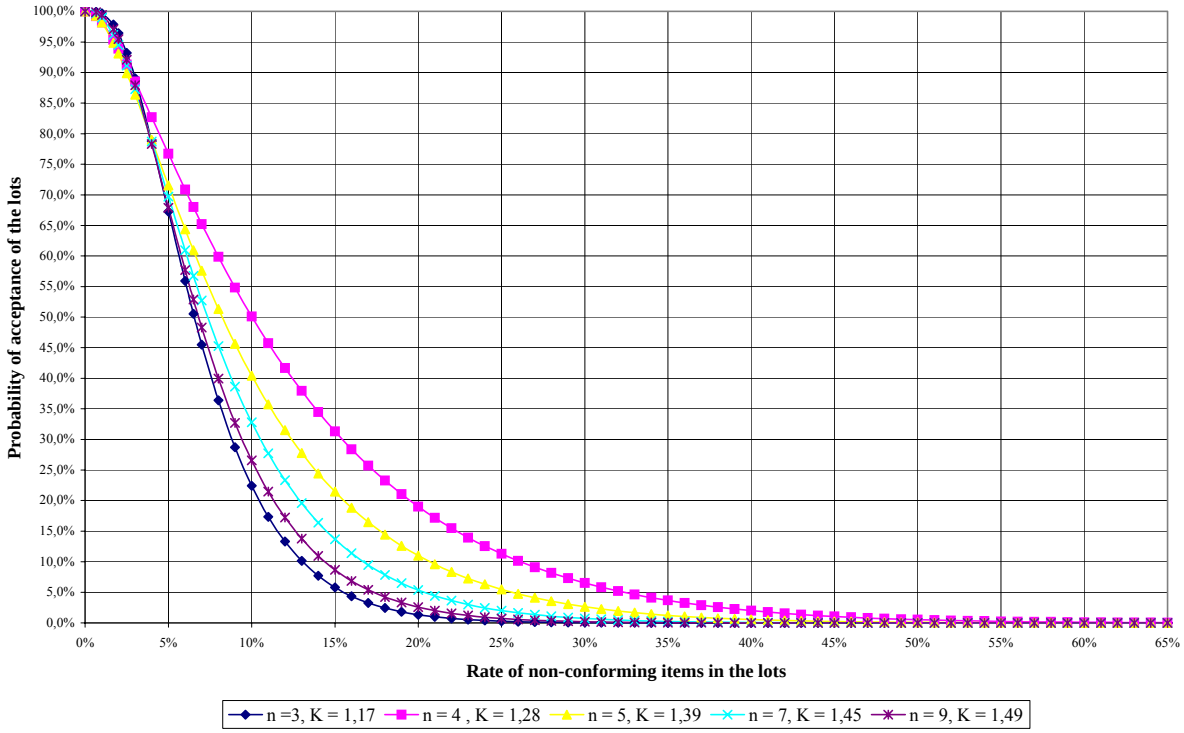
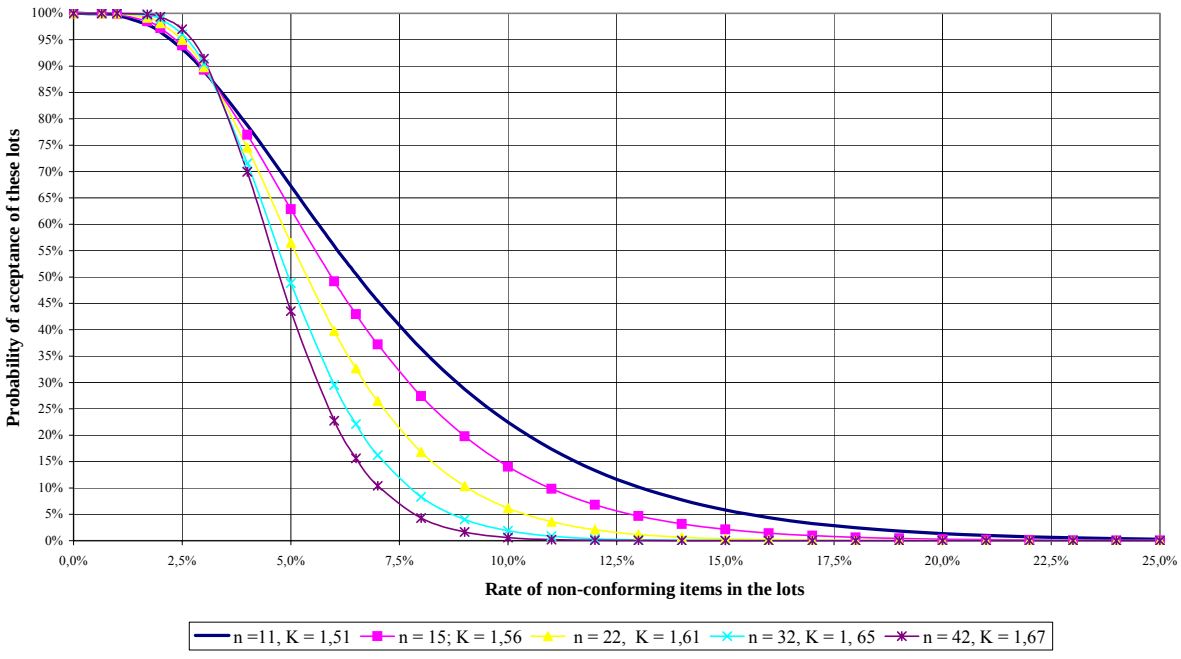


Figure 16: OC curve, variable sampling plan, σ-method, AQL = 2,5 %, n = 11 to 42



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

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

$s = \sqrt{\dfrac{\sum_{i=1}^{n} \left(x_i - \bar{x}\right)^2}{n-1}}$ 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[29].

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

Table 20: Selected t-values of the Student's distribution

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

Decision Rules

M is considered by the Codex specification as a minimum value for the mean

Example: fat content of a whole milk

The lot is accepted if:

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

and rejected otherwise.

Table 20 provides t-values of the Student's distribution for some selected sample sizes and for α of 5 % and 0,5 %.

M is considered by the Codex specification as a maximum value for the mean

Example: Sodium content of a diet rusk

The lot is accepted if:

$$\bar{x} \leq M + \frac{t_\alpha \times s}{\sqrt{n}}$$

and rejected otherwise.

M is considered by the Codex specification neither as a minimum value for the mean, neither as a maximum value for the mean

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

Example: Vitamin C content in an infant formula

The lot is accepted if

$$M - \frac{t_{\alpha/2} \times s}{\sqrt{n}} \leq \bar{x} \leq M + \frac{t_{\alpha/2} \times s}{\sqrt{n}}$$

and rejected otherwise.

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 standardardized Normal distribution, corresponding to the significance level α[30] ($u_{0,05}$ = 1,645, $u_{0,005}$ = 2,576).

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

Decision Rules

M is considered by the Codex specification as a minimum value for the mean

Example: fat content of a whole milk

The lot is accepted if:

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

and rejected otherwise.

M is considered by the Codex specification as a maximum value for the mean

Example: Sodium content of a diet rusk

The lot is accepted if:

$$\bar{x} \leq M + \frac{u_\alpha \times \sigma}{\sqrt{n}}$$

and rejected otherwise.

M is considered by the Codex specification neither as a minimum value for the mean, neither as a maximum value for the mean

Example: Vitamin C content in an infant formula

The lot is accepted if

$$M - \frac{u_{\alpha/2} \times \sigma}{\sqrt{n}} \leq \bar{x} \leq M + \frac{u_{\alpha/2} \times \sigma}{\sqrt{n}}$$

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

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

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 Procedure N° 12: Guide on Sampling for Analysis of Foods, 2002

DRAFT GUIDELINES ON MEASUREMENT UNCERTAINTY

(At Step 8 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, 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.

These guidelines only apply to quantitative analysis.

Most quantitative analytical results take the form of "a ± 2u or a ± U" where "a" is the best estimate of the true value of the concentration of the mesurand (the analytical result) and "u" is the standard uncertainty and "U" (equal to 2u) is the expanded uncertainty. The range "a ± 2u" represents a 95% level of confidence where the true value would be found. The value of "U" or "2u" is the value which is normally used and reported by analysts and is hereafter referred to as "measurement uncertainty" and may be estimated in a number of different ways.

Terminology

The international 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"[2]

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

1. The measurement uncertainty associated with all analytical results is to be estimated.
2. The measurement uncertainty of an analytical result may be estimated by 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 International vocabulary of basic and general terms in metrology, ISO 1993, 2nd Edition.

3 The measurement uncertainty and its level of confidence must, on request, be made available to the user (customer) of the results.

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.

PROPOSED DRAFT GUIDELINES FOR EVALUATING ACCEPTABLE METHODS OF ANALYSIS

(At Step 5 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 should 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 as appropriate 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
- linearity
- precision; repeatability intra-laboratory reproducibility inter-laboratory
- 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 (matrix matched and similar level of analyte) should be used to determine the accuracy of the method of analysis used.

NMKL Procedure 9 (2001)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-value.

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

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.

If the reference value is $X_{certified} \pm CI$ (95% confidence interval) then:

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

A z-value outside the range $|z| \leq 2$ indicates a significant bias and a bias correction should be made.

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 - 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 Detection Limit 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 Detection Limit 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.	

The determination limit 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 Detection Limit nor Determination Limit 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 Detection Limit.

Determination Limit– 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 Determination Limit 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 Detection Limit. 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 Determination Limit by inspection. Express Determination Limit as the lowest analyte concentration which can be determined with an acceptable level of uncertainty.
Normally Determination Limit 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 quantity 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 Determination Limit (b)		As for Determination Limit. Determination Limit 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.

$$\sigma = 0.22c \qquad if \quad c < 1.2\times10^{-7}$$
$$\sigma = 0.02c^{0.8495} \qquad if \quad 1.2\times10^{-7} \le c \le 0.138$$
$$\sigma = 0.01c^{0.5} \qquad if \quad c > 0.138$$

RECOVERY

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 is unclear whether or not interferences are already present, the selectivity of the method can 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.**]**

NMKL Procedure No. 9

ALINORM 04/27/23
APPENDIX VI

STATUS OF ENDORSEMENT OF METHODS OF ANALYSIS AND SAMPLING

Part I. METHODS OF ANALYSIS

A. Codex Committee on Fats and Oils
B. Codex Committee on Fish and Fishery Products
C. *Ad hoc* Intergovernmental Task Force on Fruit and Vegetable Juices
D. Codex Committee on Nutrition and foods for special dietetary uses
E. Methods of Analysis for Additives and Contaminants

A. CODEX COMMITTEE ON FATS AND OILS[1]

COMMODITY	PROVISION	METHOD	PRINCIPLE	NOTE	TYPE	STATUS
Fat Spreads and Blended Spreads	Milk fat content (butyric acid)	IUPAC 2.310, AOAC 990.27 or AOCS Ca 5c-87 (97).	Saponification, acidification, water soluble fatty acid separation, GLC determination	CCFO should determine a conversion factor	I	TE
Olive Oils and Olive Pomace Oils	Organoleptic characteristics	COI/T.20/Doc. no. 15.	Panel test		I	E
	Free acidity (acid value)	ISO 660:1996, amended 2003 or AOCS Cd 3d-63 (03)	Titrimetry		I	E
	Fatty acid composition	COI/T.20/Doc. no. 24 and ISO 5508: 1990 and AOCS Ch 2-91(02) or AOCS Ce 1f-96 (02) For sample preparation ISO 5509: 2000 or AOCS Ce 2-66 (97)	Gas chromatography of methyl esters		II	E
	Trans fatty acids content	COI/T.20/Doc no. 17 or ISO 15304:2002 or AOCS Ce 1f-96 (02)	Gas chromatography of methyl esters		II	E
	Wax content	COI/T.20/Doc. no. 18 or AOCS Ch 8-02 (02)	Gas chromatography		II	E

[1] ALINORM 03/17, Appendices II, III and IV

COMMODITY	PROVISION	METHOD	PRINCIPLE	NOTE	TYPE	STATUS
	Difference between the actual and theoretical ECN 42 triglyceride content	COI/T.20/Doc. no. 20 or AOCS Ce 5b-89 (97)	Analysis of triglycerides of HPLC and calculation		I	E
	Sterol composition and total sterols	COI/T.20/Doc. no. 10 or ISO 12228:1999 or AOCS Ch 6-91 (97).	Gas chromatography		II	E
	Erythrodiol + uvaol content	IUPAC 2.431.	Gas chromatography		II	E
	Stigmastadienes	COl/T.20/Doc. no. 11 or ISO 15788-1:1999 or AOCS Cd 26-96 (03).	Gas chromatography		II	E
	Stigmastadienes	ISO 15788-2: 2003	HPLC	CCFO is asked to consider whether the method is appropriate	III	TE
	Peroxide value	ISO 3960:2001 or AOCS Cd 8b-90 (03).	Titrimetry		I	E
	Absorbency in ultra-violet	COI/T.20/Doc. No. 19 or ISO 3656:2002 or AOCS Ch 5-91 (01).	Absorption in ultra violet		II	E
	Alpha-tocopherol	ISO 9936:1997	HPLC		II	E
Olive Oils and Olive Pomace Oils	Arsenic	AOAC 952.13	Colorimetry (diethyldithiocarbamate)		III	E
	Arsenic	AOAC 942.17	Colorimetry (Molybdenum blue)		III	E
	Arsenic	AOAC 986.15	AAS		II	E
	Lead	AOAC 994.02 or ISO 12193:2004 or AOCS Ca 18c-91(97)	AAS		II	E
	Traces of halogenated solvents	COI/T.20/Doc. no. 8.	Gas chromatography		II	E
	Moisture and volatile matter	ISO 662:1998	Gravimetry		I	E
	Insoluble impurities in light petroleum	ISO 663:2000	Gravimetry		I	E
	Iron and copper	ISO 8294:1994 or AOAC 990.05	AAS		II	E
	Saponification value	ISO 3657:2002 or AOCS Cd 3-25 (03)	Titrimetry		I	E
	Unsaponifiable matter	ISO 3596:2000 or ISO 18609:2000 or AOCS Ca 6b-53 (01)	Gravimetry		I	E

COMMODITY	PROVISION	METHOD	PRINCIPLE	NOTE	TYPE	STATUS
	Fatty acids in the 2-position of the triglycerides	ISO 6800:1997 or AOCS Ch 3-91 (02)	Gas chromatography		I	E
	Relative density	IUPAC 2.101, with the appropriate conversion factor	Pycnometry		I	E
	Refractive index	ISO 6320:2000 or AOCS Cc 7-25 (02)	Refractometry		II	E
	Iodine value	ISO 3961:1996 or AOAC 993.20 or AOCS Cd 1d-92 (97) or NMKL 39 (2003)	Wijs-Titrimetry		I	E

Amendments proposed to the methods in the current standard for Named Vegetable Oils

COMMODITY	PROVISION	METHODS	PRINCIPLE	TYPE	STATUS
Named Vegetable Oils	Acidity	ISO 660: 1996, amended 2003; or AOCS Cd 3d-63 (03)	Titrimetry	I	E
	Apparent density	ISO 6883: 2000, with the appropriate conversion factor; or AOCS Cc 10c-95 (02)	Pycnometry	I	E
	Arsenic	AOAC 952.13	Colorimetry (diethyldithiocarbamate)	III	E
	Arsenic	AOAC 942.17	Colorimetry (molybdenum blue)	III	E
Named Vegetable Oils	Arsenic	AOAC 986.15	AAS	II	E
	Copper and iron	ISO 8294: 1994; or AOAC 990.05; or AOCS Ca 18b-91 (03)	AAS	II	E
	Crismer value	AOCS Cb 4-35 (97) and AOCS Ca 5a-40 (97)	Turbidity	I	E
	GLC ranges of Fatty acid composition	ISO 5508: 1990 and ISO 5509: 2000; or AOCS Ce 2-66 (97) and Ce 1e-91 (01) or Ce 1f-96 (02)	Gas chromatography of methyl esters	II	E
	Insoluble impurities	ISO 663: 2000	Gravimetry	I	E
	Iodine value (IV)	Wijs - ISO 3961: 1996; or AOAC 993.20; or AOCS Cd 1d-1992 (97); or NMKL 39 (2003)	Wijs-Titrimetry [2]	I	E

[2] It is possible to calculate the Iodine Value from fatty acid composition data obtained by gas chromatography e.g. using AOCS Cd 1b-87 (97)

	Lead	AOAC 994.02 ; or ISO 12193: 2004; or AOCS Ca 18c-91 (03)	Atomic Absorption	II	E
	Moisture & volatile matter at 105°C	ISO 662: 1998	Gravimetry	I	E
	Peroxide value (PV)	AOCS Cd 8b-90 (03); or ISO 3960: 2001	Titrimetry	I	E
	Refractive index	ISO 6320: 2000; or AOCS Cc 7-25 (02)	Refractometry	II	E
	Reichert value and Polenske value	AOCS Cd 5-40 (97)	Titrimetry	I	E
	Saponification value (SV)	ISO 3657: 2002; or AOCS Cd 3-25 (03)	Titrimetry	I	E
	Slip point	ISO 6321:2002 for all oils; AOCS Cc 3b-92 (02) for all oils except palm oils; AOCS Cc 3-25 (97) for palm oils only	Open ended capillary tube	I	E
	Soap content	BS 684 Section 2.5; or AOCS Cc 17-95 (97)	Gravimetry	I	E
	Sterol content	ISO 12228: 1999; or AOCS Ch 6-91 (97)	Gas chromatography	II	E
	Tocopherol content	ISO 9936: 1997; or AOCS Ce 8-89 (97)	HPLC	II	E
	Unsaponifiable matter	ISO 3596: 2000; or ISO 18609: 2000; or AOCS Ca 6b-53 (01)	Gravimetry	I	E

B. CODEX COMMITTEE ON FISH AND FISHERY PRODUCTS[3]

Draft Standard for Salted Atlantic Herring and Salted Sprats (at Step 8)

Draft Amendment to the Standard for Quick Frozen Fish Sticks (Fish Fingers), Fish Portions and Fish Fillets – Breaded or in Batter

COMMODITY	PROVISION	METHOD	PRINCIPLE	TYPE	STATUS
Salted Atlantic Herring and Salted Sprat	Water content	AOAC 950.46B	air drying	I	E
Quick Frozen Fish Sticks	Fish content (declaration)	AOAC 996.15 and calculation (see below)	see below	I	E

Section 7. Sampling, Examination and Analysis

7.4 Estimation of Fish Content

According to AOAC Method 996.15. In cases where there is some remaining doubts over the composition of the fish core then the method of analysis as outlined below could be used, i.e. as a reference method.

Determination of Fish Content

The fish content of a fish finger (fish stick) is calculated by using the following equation

$$\%\text{Fish Content} = \frac{\text{Weight of ingoing fish}}{\text{Weight of final product}} x100$$

For most products therefore, the fish ingredient weight is that of the raw ingredient. Any figure placed or declared on a product label would be a typical quantity reflecting the producer's normal manufacturing variations, in accordance with good manufacturing practice.

Checking of fish content by chemical analysis

The percentage fish content, corrected for the non-fish flesh nitrogen contributed by the carbohydrate coating, is calculated as follows.

$$\%\text{Fish} = \frac{(\%\text{ total nitrogen} - \%\text{ non - fish flesh nitrogen}}{\text{N factor *}} x100$$

* appropriate N (nitrogen) factor

The non-fish flesh nitrogen is calculated as follows: % non-fish flesh nitrogen = % carbohydrate x 0.02

[3] ALINORM 04/27/18, Appendix II and Appendix VII (Draft Amendment to the Standard for Quick Frozen Fish Sticks (Fish Fingers), Fish Portions and Fish Fillets – Breaded or in Batter : declaration of fish content)

Where the carbohydrate is calculated by difference: % carbohydrate = 100 – (% water + % fat + % protein + % ash)

References

Determination of nitrogen: ISO 937:1978

Determination of moisture: ISO 1442:1997

Determination of total fat: ISO 1443:1973

Determination of ash: ISO 936: 1998

Table 2: Interim Nitrogen factors to be used for white fish as an ingredient (i.e. after GMP)

SPECIES	Nitrogen %
White fish:	
Cod	2.66
Minced Cod	2.61
Coley/Saithe	2.69
European Hake	2.64
Haddock	2.72
Ling	2.78
Plaice	2.46
Alaskan Pollack	2.59
Whiting	2.68
White fish mean	2.65

C. *AD HOC* INTERGOVERNMENTAL TASK FORCE ON FRUIT AND VEGETABLE JUICES[4]

1. Draft General Standard for Fruit Juices and Nectars: methods endorsed

.

COMMODITY	PROVISION	METHOD	PRINCIPLE	Type	Status
Fruit Juices and Nectars	Ascorbic acid-L (additives)	IFU Method No 17a (1995)	HPLC	II	E
	Ascorbic acid-L (additives)	ISO 6557-1: 1986	Fluorescence spectrometry	IV	E
	Ascorbic acid-L (additives)	AOAC 967.21 IFU Method No 17 ISO 6557-2: 1984	Indophenol method	III	E
	Carbon dioxide (additives and processing aids)	IFU Method No 42 (1976)	Titrimetry (back-titration after precipitation)	IV	E
	Cellobiose	IFU Recommendation No.4 October 2000	Capillary gas chromatography	IV	E
	Citric acid [5](additives)	AOAC 986.13	HPLC	II	E
	Citric acid [5](additives)	EN 1137: 1994 IFU Method No 22 (1985)	Enzymatic determination	III	E
	Glucose and fructose (permitted ingredients)	EN 12630 IFU Method No 67 (1996) NMKL 148 (1993)	HPLC	III	E
	Glucose-D and fructose-D (permitted ingredients)	EN 1140 IFU Method No 55 (1985)	Enzymatic determination	II	E
	Malic acid (additives)	AOAC 993.05	Enzymatic determination and HPLC	III	E
	Malic acid-D	EN 12138 IFU Method No 64 (1995)	Enzymatic determination	II	E
	Malic acid-D in apple juice	AOAC 995.06	HPLC	II	E
	Malic acid-L	EN 1138 (1994) IFU Method No 21 (1985)	Enzymatic determination	II	E
	Pectin (additives)	IFU Method No 26 (1964/1996)	Precipitation/photometry	I	E

[4] ALINORM 03/39A, Appendices II and III
[5] All juices except citrus based juices

	Benzoic acid and its salts; sorbic acid and its salts	IFU Method No 63 (1995) NMKL 124 (1997)	HPLC	II	E
	Benzoic acid and its salts	ISO 5518:1978 ISO 6560: 1983	Spectrometry	III	E
	Preservatives in fruit juices (sorbic acid and its salts)	ISO 5519: 1978	Spectrometry	III	E
	Saccharin	NMKL 122 (1997)	Liquid chromatography	II	E
	Soluble solids	AOAC 983.17 EN 12143 (1996) IFU Method No 8 (1991) ISO 2173: 2003	Indirect by refractometry	I	E
	Sucrose (permitted ingredients)	EN 12146 (1996) IFU Method No 56 (1985/1998)	Enzymatic determination	III	E
	Sucrose (permitted ingredients)	EN 12630 IFU Method No 67 (1996) NMKL 148 (1993)	HPLC	II	E
	Sulphur dioxide (additives)	Optimized Monier Williams AOAC 990.28 IFU method No. 7A (2000) NMKL 132 (1989)	Titrimetry after distillation	II	E
	Sulphur dioxide (additives)	NMKL 135 (1990)	Enzymatic determination	III	E
	Sulphur dioxide (additives)	ISO 5522:1981 ISO 5523:1981	Titrimetry after distillation	III	E
	Tartaric acid in grape juice (additives)	EN 12137 (1997) IFU Method No 65 (1995)	HPLC	II	E
	Total nitrogen	EN 12135 (1997) IFU Method No 28 (1991)	Digestion/titration	I	E

2. Draft General Standard for Fruit Juices and Nectars: methods temporary endorsed (subject to the finalisation by the Task Force of the provisions listed)

COMMODITY	PROVISION	METHOD	PRINCPLE	TYPE	STATUS
Fruit Juices and Nectars	Acetic acid	EN 12632 or IFU Method No 66 (1996)	Enzymatic determination		TE
	Alcohol (ethanol)	IFU Method No 52 (1983/1996)	Enzymatic determination		TE
	Anthocyanins	IFU Method No 71 (1998)	HPLC		TE
	Ash in fruit products	AOAC 940.26 EN 1135 (1994) - IFU Method No 9 (1989)	Gravimetry		TE
	Beet sugar in fruit juices	AOAC 995.17	Deuterium NMR		TE
	Benzoic acid as a marker in orange juice	AOAC 994.11	HPLC		TE
	Determination of C^{13}/C^{12} ratio of ethanol derived from fruit juices	JAOAC 79, No.1, 1996, 62-72	Stable isotope mass spectrometry		TE
	Carbon stable isotope ratio of apple juice	AOAC 981.09 - JAOAC 64, 85 (1981)	Stable isotope mass spectrometry		TE
	Carbon stable isotope ratio of orange juice	AOAC 982.21)	Stable isotope mass spectrometry		TE
	Carotenoid, Total/individual groups	EN 12136 (1997) - IFU Method No59 (1991)	Precipitation/fractionation		TE
	Carotenoids, Total	ISO 6558-2:1992	Column chromatographic separation and spectrometry		TE
	Centrifugable pulp	EN 12134 - IFU Method No 60 (1991/1998)y	Centrifugation/% value		TE
	Chloride (expressed as sodium chloride)	EN12133 IFU Method No 37 (1968)	Electrochemical titrimetry		TE
	Chloride in vegetable juice	AOAC 971.27 (Codex general method) ISO 3634:1979	Titration		TE

Fruit Juices and Nectars	Essential oils	AOAC 968.20 - IFU 45b	(Scott) distillation, titration		TE
	Essential oils (in citrus fruit)	ISO 1955:1982	Distillation and direct reading of the volume		TE
	Fermentability	IFU Method No 18 (1974)	Microbiological method		TE
	Formol number	EN 1133 (1994) IFU Method No 30 (1984)	Potentiometric titration		TE
	Free amino acids	EN 12742 IFU Method No 57 (1989)	Chromatography		TE
	Fumaric acid	IFU Method No 72 (1998)	HPLC		TE
	Glucose, fructose, sorbitol	EN 12630 IFU Method No 67 (1996) NMKL 148 (1993)	HPLC		TE
	Gluconic acid	IFU Method No 76 (2001)	Enzymatic determination		TE
	Glycerol	IFU Method No 77 (2001)	Enzymatic determination		TE
	Hesperidin and naringin	EN 12148 (1996) - IFU Method No 58 (1991)	HPLC		TE
	HFCS & HIS in apple juice (permitted ingredients)	JAOAC 84, 486 (2001)	CAP GC Method		TE
	Hydroxymethylfurfural	IFU Method No 69 (1996)	HPLC		TE
	Hydroxymethylfurfural	ISO 7466:1986	Spectrometry		TE
	Isocitric acid-D	EN 1139 - IFU Method No 54 (1984)	Enzymatic determination		TE
	Lactic acid- D and L	EN 12631 (1999) IFU Method No 53 (1983/1996)	Enzymatic determination		TE
	L-malic/total malic acid ratio in apple juice	AOAC 993.05			TE

	Naringin and neohesperidin in orange juice	AOAC 999.05	HPLC		TE
	pH-value	EN 1132 (1994) IFU Method No 11 (1968/1989) ISO 1842:1991	Potentiometry		TE
	Phosphorus/Phosphate	EN 1136 (1994) IFU Method No 50 (1983)	Photometric determination		TE
	Proline	EN 1141 (1994) IFU Method No 49 (1983)	Photometry		TE
	Quinic acid in cranberry juice cocktail and apple juice	AOAC 986.13	HPLC		TE
	Recoverable oil	AOAC 968.20 - IFU Method No 45b	Distillation and titration Scott method		TE
	Relative density	EN 1131 (1993) IFU Method No 1 (1989) & IFU Method No General sheet (1971)	Pycnometry		TE
	Relative density	IFU Method No 1A	Densitometry		TE
	Sodium, potassium, calcium, magnesium	EN 1134 (1994) IFU Method No 33 (1984)	Atomic Absorption Spectroscopy		TE
	Sorbitol-D	IFU Method No 62 (1995)	Enzymatic determination		TE
	Stable carbon isotope ratio in the pulp of fruit juices	ENV 13070 (1998) Analytica Chimica Acta 340 (1997)			TE
	Stable carbon isotope ratio of sugars from fruit juices	ENV 12140 Analytica Chimica Acta.271 (1993)	Stable isotope mass spectrometry		TE
	Stable hydrogen isotope ratio of water from fruit juices	ENV 12142 (1997)	Stable isotope mass spectrometry		TE
	Stable oxygen isotope ratio in fruit juice water	ENV 12141(1997)	Stable isotope mass spectrometry		TE
	Starch	AOAC 925.38 IFU Method No 73	Precipitation		TE

	Sugar -beet derived syrups in frozen concentrated orange juice $\delta^{18}O$ Measurements in water	AOAC 992.09	Oxygen isotope ratio analysis		TE
	Titrable acids, total	EN 12147 (1995) IFU Method No Method No 3, (1968) ISO 750:1998	Titrimetry		TE
	Total dry matter	EN 12145 (1996) IFU Method No 61 (1991)	Gravimetric determination		TE
	Total solids	AOAC 985.26	Microwave oven drying		TE
	Vitamin C	AOAC 967.22	Microfluorometry		TE
	Vitamin C	CEN/TC275/WG9 N60	DNA		TE

D. CODEX COMMITTEE ON NUTRITION AND FOODS FOR SPECIAL DIETARY USES

Draft Revised Standard for Gluten-Free Foods (At Step 7)

COMMODITY	PROVISION	METHOD	PRINCIPLE	NOTE	TYPE	STATUS
Gluten-free foods	Gluten	Enzyme-Linked Immunoassay R5 Mendez (ELISA) Method	Immunoassay	CCFNSDU to provide clarification on the application of the Method	IV	TE

E. METHODS OF ANALYSIS FOR ADDITIVES AND CONTAMINANTS

1. CONTAMINANTS

Amendment to the methods endorsed by the 23rd Session of the CCMAS and adopted in 2001 (CODEX STAN 228-2001), as a result of the update of the methods for lead, copper and iron in fats and oils.

COMMODITY	PROVISION	METHOD	PRINCIPLE	TYPE	STATUS
All foods (except fats and oils)	Lead, cadmium, copper, iron and zinc	NMKL 139 (1991) AOAC 999.11	AAS after dry ashing	II	E
All foods (except fats and oils)	Lead, cadmium, copper, iron and zinc	NMKL 161 (1998) AOAC 999.10	AAS after microwave digestion	III	E

Amendments to the current list of methods

COMMODITY	PROVISION	METHOD	PRINCIPLE	NOTE	TYPE	STATUS
All foods	Cadmium	AOAC 986.15	Anodic stripping voltametry		III	E
All foods	Copper	AOAC 960.40	Colorimetry (diethyldithiocarbamate)		III	E
All foods	Lead	AOAC 972.25	AAS	Type II method adopted in 2001 (see above)	III	E
All foods except fats and oils	Lead	AOAC 982.23	Anodic stripping voltametry		III	E
All foods	Lead	AOAC 986.15	Anodic stripping voltametry		III	E
All foods	Zinc	AOAC 969.32	AAS	Type II method adopted in 2001 (see above)	III	E
All foods	Zinc	AOAC 986.15	AAS		III	E
~~Fats and oils~~	~~Nickel~~	~~IUPAC 2.631~~ ~~AOAC 990.05~~ ~~ISO 8294:1994~~	~~Atomic absorption spectrometry (direct graphite furnace)~~	Deleted as no provisions exist	~~II~~	E

2. ADDITIVES

COMMODITY	PROVISION	METHOD	PRINCIPLE	NOTE	TYPE	STATUS
Meat Products	Nitrates and/or Nitrites	EN 12014-3:1998-06 Part 3	Spectrometric determination of nitrate and nitrite content of meat products after enzymatic reduction of nitrate to nitrite	TE at the 24th session	III[6]	E
Meat Products	Nitrates and/or Nitrites	NMKL 165 (2000) EN 12014-4:1998-06 Part 4	Ion-exchange chromatographic method	TE at the 24th session	III	E

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

Amendments to the current list of methods for additives

COMMODITY	PROVISION	METHOD	PRINCIPLE	NOTE	TYPE	STATUS
Beverages and sweets (including fruits juices)	Saccharin	NMKL 122 (1997)	Liquid chromatography	Endorsed in 2003 Subject to finalization of relevant provisions for saccharin	II[7]	E
Fats and oils	Butylhydroxyanisole, butylhydroxytoluene, tert-butylhydroquinone, ~~nordihydroguaiaretic acid~~ & propyl gallate	AOAC 983.15	Liquid chromatography	nordihydroguaiaretic acid deleted as no provision exist	II	E

Part II. SAMPLING

CODEX COMMITTEE ON FATS AND OILS (ALINORM 03/17, Appendix II)

Standard for Olive Oils and Olive-Pomace Oils

Sections 8.16 and Annex-Section 4.12 Sampling

According to ISO 661:1989 and ISO 5555:2001.

[7] Inclusion in the final list subject to finalization of provisions for saccharin in the Draft Standard for Fruit Juices and Nectars or in the General Standard for Food Additives

THE USE OF ANALYTICAL RESULTS: SAMPLING PLANS, RELATIONSHIP BETWEEN THE ANALYTICAL RESULTS, THE MEASUREMENT UNCERTAINTY, RECOVERY FACTORS AND PROVISIONS IN CODEX STANDARDS

ISSUES INVOLVED

There are a number of analytical and sampling considerations which prevent the uniform implementation of legislative standards. In particular, different approaches may be taken regarding sampling procedures, the use of measurement uncertainty and recovery corrections.

At present there is no official guidance on how to interpret analytical results across the Codex Community. Significantly different decisions may be taken after analysis of the "same sample". For example some countries use an "every-item-must-comply" sampling regime, others use an "average of a lot" regime, some deduct the measurement uncertainty associated with the result, others do not, some countries correct analytical results for recovery, others do not. This interpretation may also be affected by the number of significant figures included in any commodity specification.

It is essential analytical results are interpreted in the same way if there is to be equivalence across the Codex Community.

It is stressed that this is not an analysis or sampling problem as such but an administrative problem which has been highlighted as the result of recent activities in the analytical sector, most notably the development of International Guidelines on the Use of Recovery Factors when Reporting Analytical Results and various Guides prepared dealing with Measurement Uncertainty.

RECOMMENDATIONS

It is recommended that when a Codex Commodity Committee discusses and agrees on a commodity specification and the analytical methods concerned, it states the following information in the Codex Standard:

1. Sampling Plans

The appropriate sampling plan to control conformity of products with the specification. This should state:

- whether the specification applies to every item in a lot, to the average in a lot or the proportion non-conforming;
- the appropriate acceptable quality level to be used;
- the acceptance conditions of a lot controlled, in relation to the qualitative/quantitative characteristic determined on the sample.

2. Measurement Uncertainty

That an allowance is to be made for the measurement uncertainty when deciding whether or not an analytical result falls within the specification. This requirement may not apply in situations when a direct health hazard is concerned, such as for food pathogens.

3. Recovery

[Where relevant and appropriate the analytical results are to be reported on a recovery corrected basis and that the recovery should be quoted in any analytical report.]

4. Significant Figures

The units in which the results are to be expressed and the number of significant figures to be included in the reported result.