## codex alimentarius commission

FOOD AND AGRICULTURE ORGANIZATION OF THE UNITED NATIONS

WORLD HEALTH ORGANIZATION

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**ALINORM 99/23** 

#### JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX ALIMENTARIUS COMMISSION
Twenty-third Session
Rome, 28 June – 3 July 1999

REPORT OF THE TWENTY-SECOND SESSION OF THE CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING Budapest, Hungary, 23 – 27 November 1998

**Note:** This report includes Codex Circular Letter CL 1998/42-MAS.

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CX 4/50.2

CL 1998/42-MAS

December 1998

**TO:** Codex Contact Points

**Interested International Organizations** 

**FROM:** Chief, Joint FAO/WHO Food Standards Programme

FAO, Viale delle Terme di Caracalla, 00100 Italy

SUBJECT: DISTRIBUTION OF THE REPORT OF THE TWENTY-SECOND SESSION OF THE CODEX

COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING (ALINORM 99/23)

The report of the Twenty-second Session of the Codex Committee on Methods of Analysis and Sampling (CCMAS) is attached. It will be considered by the Twenty-third Session of the Codex Alimentarius Commission (Rome, 28 June – 3 July 1999).

## PART A MATTERS FOR CONSIDERATION BY THE 23RD SESSION OF THE CODEX ALIMENTARIUS COMMISSION

#### 1. Methods of Analysis and Sampling

- (i) Method of Analysis Provisions of Certain Commodity Standards (ALINORM 99/23, Appendix III, Part 1)
- (ii) Method of Sampling Provisions of Certain Commodity Standards (ALINORM 99/23, Appendix III, Part 2)

Governments wishing to propose amendments or to submit comments regarding the implications which the above matters have for their economic interests should do so in writing, in conformity with the *Codex Alimentarius Commission Procedural Manual*, to the Chief, Joint FAO/WHO Food Standards Programme, FAO, Via delle Terme di Caracalla, 00100 Rome, Italy, **no later than 15 April 1999**.

### PART B REQUEST FOR COMMENTS ON PROPOSED DRAFT AMENDMENTS TO THE PROCEDURAL MANUAL AT STEP 3

Governments and interested international organizations are invited to comment on the Proposed Draft Amendments to the Procedural Manual, as contained in Appendix II of this report, at Step 3. Comments should be sent to the Chief, Joint FAO/WHO Food Standards Programme, FAO, Via delle Terme di Caracalla, 00100 Rome, Italy, **no later than 15 October 1999**.

#### SUMMARY AND CONCLUSIONS

The Twenty-second Session of the Codex committee on Methods of Analysis and Sampling reached the following conclusions:

#### **Matters for Consideration by the Commission**

#### The Committee:

- proposed to the Commission that new work be initiated on the amendments of the relevant sections of the Codex Alimentarius Commission Procedural Manual in order to enable the implementation of the criteria approach by this Committee (para. 28 & Appendix II); and
- endorsed a number of methods of analysis and sampling for 17 Codex commodity standards and the Guidelines for Nutrition Labelling (paras 53-59 & Appendix III).

#### Other Matters of Interest to the Commission and Other Codex Committees

#### The Committee:

- returned the Proposed Draft Guidelines on Sampling to Step 3 for further redrafting (1) taking into consideration comments as appropriate; (2) making the text easier, simpler and more user-friendly; (3) incorporating a new explanatory note which elucidates what is a "sampling plan" and which kind of sampling plan is to be used for the specific control to be performed; and incorporating worked examples for specific cases (paras 9-13);
- confirmed its previous general acceptance of the criteria approach for methods of analysis for chemical entities and decided to proceed with the implementation of the criteria approach including the preparation of guidelines on the application of criteria approach by this Committee and amendments of the relevant sections of the *Codex Alimentarius Commission Procedural Manual* (paras 19-28; see also the above);
- requested that when the *Harmonized Guidelines for the Use of Recovery Factors in Analytical Measurements* was published by IUPAC, the text of the Guidelines should be circulated to member countries of the Commission by way of a Codex circular letter for comments in order for this Committee to decide at its next Session whether or not to recommend the text to the Commission for adoption by reference (paras 32-35);
- agreed that a paper should be prepared on the need and definitions of measurement limits in relation to the Analytical Terminology for Codex Use for consideration at its next Session (paras 36-40);
- decided to defer further discussion on measurement uncertainty pending the publication of the *EURACHEM Guide on Measurement Uncertainty* and to request a paper on the relationship between the analytical result, measurement uncertainty and specification in Codex standards (paras 41-46, 71);
- decided to request a paper on the use of information from the proficiency testing studies for the elaboration of characteristics of in-house validated methods for consideration at its next Session and agreed that when the next draft of the *Harmonized Guidelines for the In-house Validation of Methods of Analysis* became available by IUPAC, it would consider the text to determine its suitability for Codex purposes (paras 47-51);
- agreed that it would have no objection to the use of proprietary methods, provided that similar methods or materials supplying similar results were available (para. 8);

- agreed to ask Codex commodity committees to provide information as required by the Checklists contained in the Codex Alimentarius, Volume 13, and the *Codex Alimentarius Commission Procedural Manual*, when they send methods of analysis and sampling to this Committee for endorsement (para. 60);
- recommended that commodity committees should select methods from the existing Codex general methods wherever possible and use the SI unit system in the specifications of Codex standards (paras 61-62);
- agreed to refer back to the Codex Committee on Processed Fruits and Vegetables the question regarding tolerances permitted for the declaration of net drained weight as it felt that the issue was rather a technological problem and that it would not seem feasible to establish general tolerances for net drained weight (para. 6);
- agreed that information should be sought from commodity committees on the acceptance of the statistical approach to sampling when defining compliance with the specifications in Codex standards (para. 12);
- agreed to refer the annex of CX/MAS 98/5 concerning trade dispute situations to the Codex Committee on food Import and Export Inspection and Certification System for consideration (paras 29-31);
- felt it inappropriate to combine the Draft Revised Recommended Methods of Sampling for the Determination of Pesticide Residues for Compliance with MRLs developed by the Codex Committee on Pesticide Residues (CCPR) with the Proposed Draft Guidelines on Sampling as they were based on two different approaches; and agreed to forward all written and oral comments on the former text to the CCPR for consideration (paras 14-18); and
- noted the report of the 13th Inter-Agency Meeting (paras 64-69)

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**ALINORM 99/23** 

## REPORT OF TWENTY-SECOND SESSION OF THE CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING Budapest, 23-27 November 1998

#### INTRODUCTION

1. The Codex Committee on Methods of Analysis and Sampling held its Twenty-second Session in Budapest, Hungary, from 23 – 27 November 1998, at the kind invitation of the Government of Hungary. Professor Péter Biacs, Director-General of the Central Food Research Institute (KÉKI) chaired the Session. The Session was attended by 114 delegates and observers from 35 Member Countries and 10 international organizations. A complete list of participants is given in Appendix I.

#### **OPENING OF THE SESSION**

2. The Session was opened and welcomed by Dr. Károly Tamás, State Secretary of Ministry of Agriculture and Rural Development. He emphasized the importance of Codex standards in the international food trade under the World Trade Organization Agreements and for the exportation of agricultural commodities from Hungary. He informed the delegates of the active participation of Hungary in Codex work and of the reorganization of the Hungarian National Codex Committee to further strengthen it by involving specialists from as wide a range as possible.

#### **ADOPTION OF THE AGENDA (Agenda Item 1)**

3. The Committee adopted the Provisional Agenda as presented in CX/MAS 98/1 with the understanding that it would consider Agenda Item 4(b) "Draft Revised Recommended Methods of Sampling for the Determination of Pesticide Residues for Compliance with MRLs" prior to Agenda Item 4(a) "Proposed Draft General Guidelines on Sampling". It also agreed to consider the endorsement of sampling methods/plans contained in CX/MAS 98/9 under Agenda Item 4 (a).

#### **APPOINTMENT OF RAPPORTEUR (Agenda Item 2)**

4. The Committee decided to proceed without a rapporteur.

#### MATTERS REFERRED TO THE COMMITTEE<sup>1</sup> (Agenda Item 3)

5. The Committee was informed of the matters arising from the 22nd Session of the Codex Alimentarius Commission and other Codex Committees. In addition, the Committee discussed the following issues.

#### Tolerances Permitted for the Declaration of Net Drained Weight

6. The Committee noted that the Codex Committee on Processed Fruits and Vegetables (CCPFV) at its 19th Session requested advice from this Committee on the tolerances permitted for the declaration of net drained weight. The Committee recalled that it had once discussed this issue without reaching consensus. Noting that this was rather a technological problem and it would not seem feasible to establish general tolerances for net drained weight, the Committee **agreed** to send the issue back to the CCPFV.

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<sup>&</sup>lt;sup>1</sup> CX/MAS 98/2; CX/MAS 98/2-Add. 1 (CRD 4)

#### **Proprietary Methods**

7. The Committee also noted the request from the 21st Session of the Codex Committee on Nutrition and Foods for Special Dietary Uses (CCNFSDU) to this Committee to consider the use of proprietary methods. The CCNFSDU noted that in some cases a proprietary method was the most specific way to detect an analyte, such as in the case of gluten detection. The Committee recalled that at its last Session it had endorsed one proprietary method, "Phadebas method", for the determination of diastase activity in honey with a note that other commercially available calibrated substrate preparations can also be used.

8. The Delegation of Sweden, supported by that of Finland, requested the Committee to consider the endorsement of an enzyme immunoassay method for gluten determination in food, as they felt that an appropriate method for gluten determination was urgently needed. However, the Committee was of the opinion that the CCNFSDU should first agree to include the above-mentioned method in the Standard for Gluten-Free Foods and to forward the method to this Committee for endorsement. The Committee **agreed** that it would have no objection to the use of proprietary methods, provided that similar methods or materials supplying similar results were available.

#### METHODS OF SAMPLING (Agenda Item 4)<sup>2</sup>

#### PROPOSED DRAFT GENERAL GUIDELINES ON SAMPLING (Agenda Item 4a)<sup>3</sup>

- 9. Since its 19th Session in 1994, the Committee had considered General Guideline on Sampling which should be applicable to all commodities. At the last Session, the Committee had decided to return the Proposed Draft Guidelines on Sampling to Step 3 for further revision and agreed to a number of elements to be included in the revision. The Committee recalled that this text was intended to replace all previous texts on sampling recommended by the Committee including the Sampling Plans for Prepackaged Foods (AQL 6.5). The Proposed Draft Guidelines had been revised by a Codex Consultant with assistance offered by Australia, Austria, Canada, Czech Republic, France, Hungary, India, Netherlands, Thailand, United Kingdom and United States.
- 10. While the current text was recognized to be a significant improvement from the previous version and to contain useful information, many delegations were generally of the opinion that it still was very complicated and difficult to understand and therefore it required revision to make it easier for both government officials and Codex commodity committees. For that purpose some delegations proposed to prepare a brief explanatory note elucidating what was a "sampling plan" in a simple manner and which kind of sampling plan was to be used according to the control to be performed. The Delegation of France offered to prepare the explanatory note with assistance provided by Australia, Netherlands, United Kingdom, United Stated and IDF.
- 11. On the question of whether the Guidelines should be based on the statistical approach as currently drafted or the pragmatic approach as in the case of the sampling methods for the determination of residues of pesticides and veterinary drugs, a majority of the delegations were in favour of the statistical approach as being scientifically defendable. (see para. 15)

Although the discussion on the endorsement on sampling (CX/MAS 98/9) took place under Agenda Item 4(a), its report is included under Agenda Item 10, Endorsement of Methods of Analysis Provisions in Codex Standards

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CX/MAS 98/3; CX/MAS 98/3-Add. 1 (comments from Argentina, Cuba, Slovak Republic, New Zealand and the United States); CX/MAS 98/3-Add. 2 (CRD 5; comments from the Codex Secretariat, France, New Zealand and Spain); CRD 6 (Comments from Finland and Hungary); CRD 10 (summary of the revision of the Guidelines and summary of comments submitted); CRD 11 (comments from Argentina)

12. The Committee **decided** to defer detailed discussions on the text due to its complexity. However, it **agreed** that the text should be revised with an objective to make it easier, simpler and more user-friendly by using appropriate structure and wording. The Committee **requested** the above mentioned countries to undertake this task as well. It reiterated its decision of the last Session that a new text should contain worked examples for specific cases to facilitate the use of the text. As to where these worked examples should be obtained, the Delegation of Hungary offered to provide some of them. It was pointed out that experts in specific commodities in those countries participating in the drafting could also contribute. The Committee **agreed** that specific attention should be given to matters relating to "heterogeneity" in bulk materials. The Committee further **agreed** that information should be sought from Codex commodity committees on the acceptance of the statistical approach to sampling when defining compliance with the specifications established in Codex standards.

#### Status of the Proposed Draft Guidelines on Sampling

13. The Committee **returned** the Proposed Draft Guidelines on Sampling to Step 3 of the Codex Procedure for redrafting by France in collaboration of Australia, Netherlands, United Kingdom, United States and IDF, including the preparation of an explanatory note, taking into consideration comments provided as appropriate. The Committee **requested** that modern technologies should be utilized for timely development of the text for distribution for comments in six months' time. The Committee **agreed** to examine the new text in depth at its next Session.

## DRAFT REVISED RECOMMENDED METHODS OF SAMPLING FOR THE DETERMINATION OF PESTICIDE RESIDUES FOR COMPLIANCE WITH MRLs (Agenda Item 4b)<sup>4</sup>

- 14. The Committee was informed that the Codex Committee on Pesticide Residues (CCPR) had started the revision of the Recommended Methods of Sampling for the Determination of Pesticide Residues at its 28th Session in 1996, and at its 30th Session (1998) had agreed to advance the amended Draft Revised Methods to Step 8 for adoption by the Commission. In order to promote harmonization within Codex, it had also agreed to bring the text to the attention of this Committee for consideration. A brief oral report on the opinion of the Codex Committee on Residues of Veterinary Drugs in Foods on the Draft Revised Recommended Methods was provided by the Secretariat<sup>5</sup>.
- 15. It was recognized that the referenced Methods of Sampling and the Guidelines on Sampling being developed by this Committee were based on two different approaches; the former on the practical approach for economic reasons, and the latter on the statistical approach (para. 11). Therefore, it was **felt** inappropriate to combine these two documents. However, it was stated that the CCPR Sampling document should not contain any contradiction to the Guidelines on Sampling.
- 16. A number of delegations stressed the need to harmonize those terms used in the document to internationally agreed ones, such as ISO 7002.
- 17. Other comments included: (1) Table 2 should be clarified to indicate that for plant products composite samples were prepared whereas for each animal product a single primary sample was taken; (2) Table 2 indicated that where the incidence of violative residues in the lot was below 5%, the number of samples to be taken would be unrealistic; (3) the procedure contained in Section 4.4 for the evaluation of results was too complex.
- 18. The Committee **agreed** to forward all written and oral comments to the CCPR for consideration.

<sup>&</sup>lt;sup>4</sup> CX/MAS 98/4, CX/MAS 98/4-Add.1 (comments from the United States), and CRD 12 (comments from Argentina).

<sup>&</sup>lt;sup>5</sup> ALINORM 99/31, paras 10-11.

## CRITERIA FOR EVALUATING ACCEPTABLE METHODS OF ANALYSIS FOR CODEX PURPOSES (Agenda Item 5)<sup>6</sup>

19. The Committee recalled that it had first considered this issue formally at its 19th Session. The Committee at its 20th Session (1995) had accepted the criteria-based approach in principle and agreed to draw up detailed working guidelines for its operation including the definitions and selection of the criteria to be used. The Committee at its last session had agreed that the paper should be revised by the Delegations of Canada, France and the United Kingdom and that trade dispute situations should also be addressed. The Committee agreed to discuss matters regarding trade dispute situations separately from the criteria approach at its present Session.

- 20. The Delegation of the United Kingdom presented the referenced paper<sup>7</sup> and explained that the objective of the criteria approach was to simplify the endorsement procedure for methods of analysis for chemical entities, and to provide for flexibility in selecting methods of analysis for such analytes. It was stated that the quality standard of analytical laboratories and the way in which they operate had dramatically changed due to quality assurance systems such as proficiency testing and accreditation. He stressed that the criteria approach was not intended to downgrade methods of analysis. Given the current confusion caused by the existence of multiple Type III methods, the Delegation urged the Committee to proceed with the criteria approach and proposed amendments to the relevant sections of the *Codex Alimentarius Commission Procedural Manual* in order for this Committee to implement the criteria approach.
- 21. The Committee **confirmed** its previous general acceptance of the criteria approach for methods of analysis for chemical entities. A majority of delegations wished to proceed with the approach. A number of delegations stated that only methods validated through inter-laboratory studies should be used and that analytical laboratories should be operating under quality assurance systems and/or good laboratory practices.
- 22. Many delegations expressed their preference of the criteria approach for flexibility it provided, in respect of the selection of methods or the availability of equipment. It was also stated that in order to achieve better results of analyses, it was desirable to be allowed to use those methods in which analysts were experienced. The Delegation of Ireland presented their experience in using both official methods and the criteria approach. It was stated that, if backed up by an appropriate quality assurance system, the criteria approach was found suitable to be used also in court. The Delegation stated that in the future more labs would seem to use the criteria approach.
- 23. Some delegations proposed that the Codex method Types II and III should be combined to provide for more flexibility. However, several other delegations were in favour of retaining the Type II classification stating the usefulness of these reference methods in relation to trade disputes or for use in the validation of alternative (automated) methods or calibration of new methods. The Committee for the time being did not wish to change the method classification.
- 24. Concerns were expressed on the number and selection of criteria to be used in this approach. The Committee recalled that it had already agreed that this Committee would convert methods proposed by the commodity committee into criteria. Some delegations and observers questioned the need for nine criteria which could lead to a possibility to exclude appropriate methods from selection for Codex purposes due to trivial criteria (see para. 66) or could necessitate redesigning of collaborative studies. Some delegations proposed to change the terms for certain criteria and to identify some of them as optional.

<sup>&</sup>lt;sup>6</sup> CX/MAS 98/5, CRD 13 (comments from Argentina), CRD 17 (comments from Russia); CRD 20 (comments from AOAC International); comments from Brazil.

<sup>&</sup>lt;sup>7</sup> Except for its Annex IV.

25. The Committee noted that some organizations of the Inter-Agency Meeting has included performance characteristics in the methods or appendices thereof, while some others had not done so. If required by this Committee, other organizations would place the information in the methods themselves. (see para. 65)

26. The Committee **decided** to proceed with the implementation of the criteria approach including amendments of the relevant sections of the *Codex Alimentarius Commission Procedural Manual*. (see below)

## Guidelines on the Application of the Criteria Approach by the Codex Committee on Methods of Analysis and Sampling

27. In accordance with the decision above, the Committee **decided** to prepare working guidelines for this Committee for implementation of the criteria approach. The Committee **requested** the United Kingdom, together with Canada, Australia, Finland, France, Germany, Netherlands, Norway, United States and the Codex Secretariat, to prepare a draft of the guidelines for consideration at its next Session. In drafting the document the content of the *Recommendations for a Checklist of Information Required to Evaluate Methods of Analysis and Sampling for Endorsement*<sup>8</sup> should be taken into consideration. If the paper was to contain examples, they should be drafted in such a way that they would provide practical instructions on the implementation of the criteria approach.

#### Amendments of the Relevant Sections of the Codex Alimentarius Commission Procedural Manual

28. The Committee **agreed** to seek approval of the Commission to initiate work on the amendments of the sections of the *Codex Alimentarius Commission Procedural Manual*, "Principles for the Establishment of Codex Methods of Analysis" and "Relations between Commodity Committees and General Committees – Methods of Analysis and Sampling". It also **agreed** to request comments from Member countries on the text as contained in page 7 of CX/MAS 98/5 at Step 3. However, as the Delegation of the United States strongly opposed to include "Type II" method in the criteria approach, the Committee **decided** to place the term "II and" in square brackets for future consideration. The text as amended is attached to this Report as Appendix II.

#### Dispute Situations

- 29. The Delegation of France presented Annex IV of the referenced paper and recalled that at the last Session the Delegations of the United States and France had expressed concerns that how to deal with trade dispute situations had not been fully addressed in CX/MAS 97/3. The Delegation explained that the annex included all possible trade dispute situations envisaged. The settlement procedure started with the comparison of the results of the export laboratory and import laboratory. If no agreement was reached in this phase, the two laboratories should first agree to the method to be used for a new analysis. If no agreement was yet obtained after the second analysis, they should take new samples according to the procedure specified in the annex. Further settlement would involve an arbitrating laboratory. The Delegation also mentioned other conditions such as quality assurance of the laboratory and archives of samples.
- 30. Many delegations highly appreciated the annex for its illustration of all possible scenarios. However, the Delegation of the United States stated that within its governmental system, it would not be possible to delegate authority to third parties.
- 31. Recognizing that the Codex Committee on Food Import and Export Inspection and Certification System is the Committee which deals with horizontal issues relating to food import and export, the Committee **agreed** to refer Annex IV of CX/MAS 98/5 to that Committee.

8 *Codex Alimentarius*, Volume 13.

# HARMONIZATION OF REPORTING OF TEST RESULTS CORRECTED FOR RECOVERY FACTORS – PROGRESS REPORT ON DEVELOPMENT OF HARMONIZED GUIDELINES FOR THE USE OF RECOVERY FACTORS IN ANALYTICAL MEASUREMENTS (Agenda Item 6) 9

- 32. The Committee recalled that it had first considered the concept of recovery factors in analytical work at its 19th Session. At its last Session it had received a progress report on the development of the Harmonized Guidelines and agreed that it be kept informed of progress being made by IUPAC in the development of the Guidelines. It had also agreed that once the Guidelines were finalized by IUPAC, the Committee would consider whether or not to recommend the Guidelines for Codex purposes.
- 33. The Delegation of the United Kingdom reported that the *Harmonized Guidelines for the Use of Recovery Information in Analytical Measurement* had been finalized and would be published before long. The finalized text was essentially the same as that contained in the annex of CX/MAS 98/6 with some editorial amendments. He explained that the issue was of concern due to differences from country to country in the application, or otherwise, of correction of analytical results, which might lead to trade disputes. For example, the corrected and uncorrected results of an analysis of the same sample could indicate that the product analyzed was in conformity with the specification in one analysis report while not in conformity in the other.
- 34. The Committee was generally of the view that there was disharmony in the use of recovery factors in the food analytical community and that it would be extremely difficult for it to reach consensus. On the need for correcting analytical results, some delegations stated that results should be corrected unless there were specific reasons not to do so. However, some other delegations were of the opinion that results were not corrected normally unless it was required to do so. Examples of uncorrected results were those of pesticide residue analyses and those obtained using Type I methods. It was stated that the conversion between corrected and uncorrected results was possible through the use of correction factors and that the report of analysis should give necessary information on the correction factor(s). It was also stated that information on recovery should be included in the method description, thereby referring to the method in the report of analysis would clarify whether or not the result had been corrected and provide for information necessary for conversion.
- 35. The Committee **decided** to postpone further discussions pending the publication of the Harmonized Guidelines. It **requested** that the Guidelines, when published by IUPAC, be circulated to Member countries and that comments on the Guidelines be sought by way of a Codex Circular Letter which would include pertinent elements of CRD 8. The Committee would consider the published Guidelines and comments submitted on the Guidelines at its next Session to decide whether it would be appropriate to recommend the document for adoption by reference by the Commission for Codex purposes.

## HARMONIZATION OF ANALYTICAL TERMINOLOGY IN ACCORDANCE WITH INTERNATIONAL STANDARDS – REPORT OF INTER-AGENCY MEETING ON "LIMITS" (Agenda Item 7) $^{10}$

36. The Committee recalled that at its last Session it had decided to send the definitions of analytical terms, not including those of "limit(s)", to the Commission, which subsequently endorsed them. It had further decided to request the Inter-Agency Meeting to recommend whether it would be appropriate to include "limits" in the selected terminology and to elaborate their definitions. A Codex Circular Letter had been sent to Member countries and international organizations requesting comments on the inclusion of the definitions of "limits" in the list of terminology, to which only few responses had been received.

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OX/MAS 98/6, CRD 8 (comments from USA); CRD 14 (comments from Argentina).

CRD 1 (Report of the Inter-Agency Meeting on Measurement Limits)

37. The Inter-Agency Meeting had considered this issue and concluded that there was no consensus view in the analytical community as to the procedure for determining and defining measurement limits although it had acknowledged the need to address the issue of measurement limits. The Inter-Agency Meeting had recognized that both this Committee and the IUPAC/AOAC/ISO Harmonization Programme were addressing the issue of in-house method validation, one aspect of which would be the establishment of measurement limits (see paras 48-51). It had recommended that future drafts of the IUPAC/AOAC/ISO Guidelines on In-House Methods Validation would address the issue with a view to developing a consensus approach not only within the food sector but across the analytical community.

- 38. Some delegations confirmed that there were a range of definitions established at the international level but no consensus had been reached on them. It was stated that among these definitions some were not applicable to the area of food analysis.
- 39. The Committee was generally of the view that this Committee should harmonize the definitions of "limits" for Codex purposes in the future as (1) there had already been the definitions of limit of detection, limit of quantitation, and limit of determination separately developed by the Codex Committees on Residues of Veterinary Drugs in Foods (CCRVDF) and on Pesticide Residues (CCPR); and (2) there would be a number of definitions considered in relation to the criteria approach and inhouse method validation (see paras 24, 37, 48-51). Some delegations stated that there was also a need for harmonization within the analytical community at the international level.
- 40. The Committee **agreed** that the United States in collaboration with Finland, France and Spain would prepare a document on this issue, including definitions as appropriate, for consideration by the Committee at its next Session. France and Spain were also to prepare the French and Spanish versions of the paper. The Committee was informed that CCPR and CCRVDF would be informed of the differences between the definitions of certain limits developed by these Committees.

#### MEASUREMENT UNCERTAINTY (Agenda Item 8) 11

- 41. The Committee recalled that the issue had first been considered at the its last Session where it had agreed to a number of recommendations and requested the Delegation of the United Kingdom to redraft the paper for consideration at this Session.
- 42. The Delegation of the United Kingdom presented the referenced paper and expressed concern that the approach developed by ISO<sup>12</sup>, and required by a number of accreditation agencies, for the calculation of measurement uncertainty would impose significant additional work and expenditure on food analysis laboratories. He informed the Committee that a project of the UK Ministry of Agriculture, Fisheries and Food revealed that in most cases similar uncertainty values were obtained from the collaborative trial data (top-down) and ISO (component-by-component or bottom-up) approaches as outlined in Table 1 of the document CX/MAS 98/7.
- 43. Most of the delegations who spoke welcomed the paper. There was a general agreement that the ISO approach was not suitable for food analysis laboratories, or too stringent, and that where collaborative study data were available, the use of the ISO approach should not be forced to laboratories for the calculation of measurement uncertainty. Many delegations were of the opinion that information on measurement uncertainty should be made available to customers only when requested and that it would not be made mandatory to include measurement uncertainty in the analytical report.
- 44. Delegations exchanged views on an appropriate term for "measurement uncertainty". Several delegations preferred the term "measurement reliability" as the term has positive connotations and the use of the same term as that in the ISO document<sup>12</sup>, while not using the ISO approach, would cause

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

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<sup>11</sup> CX/MAS 98/7; CRD 15 (Comments from Argentina)

confusion. However, several other delegations were in favour of the term "measurement uncertainty" as this term had already been used by a number of international organizations, including ISO, EURACHEM and NMKL.

- 45. The Delegation of Ireland informed the Committee that the revision of the *EURACHEM Guide* on *Measurement Uncertainty* was well advanced and the revised document containing many worked examples would be available in the near future. It was hoped that the final version of the text would address all concerns of this Committee. The Committee was also informed of the publications of NMKL on this matter which provided intermediate measurement of uncertainty.
- 46. The Committee **decided** to defer further discussion on this item to a future session pending the publication of the EURACHEM Guide so as to avoid duplication of the work of other international body (see paras 45 & 70-71).

#### IN-HOUSE METHOD VALIDATION (Agenda Item 9) 13

- 47. The Committee recalled that at its last Session it had considered a paper on establishing routine methods, which had been referred to it by the Codex Committee on Residues of Veterinary Drugs in Foods. The paper explained the difficulties encountered in the area of veterinary drug residue analysis in performing large scale method validation and finding appropriate validated methods. The Committee had proposed to initiate work on in-house method validation, which was approved by the Commission at its 22nd Session. The Delegations of the Netherlands and the United Kingdom had prepared a paper.
- 48. The Delegation of the Netherlands, in introducing the paper, stated that in the cases of analyses of food moving in trade, inter-laboratory recognition was important. However, where no collaboratively studied methods were available, an in-house method validation could be utilized. Among validation routes, it might be possible to utilize the following routes in an in-house validation scheme and then obtain an externally referenced method yielding acceptable results: (1) calibration using reference materials; and (2) comparison of results achieved with reference methods. It was further stated that an appropriate inter-laboratory study would give important information that might be extrapolated to other analytes and matrices using an in-house validation protocol. However, criteria to be established for such an in-house validation would be different from those for the normal method validation.
- 49. The Delegation of the United Kingdom reported that IUPAC had initiated work on the development of the *Harmonized Guidelines for the In-house Validation of Methods of Analysis* last year by the same working group that had finalized a number of protocols and guidelines such as those for collaborative studies and recovery factors. The text contained in Annex 1 of the referenced document was its first draft. He invited participants to comment on the IUPAC Guidelines. He also informed the Committee that there would be an FAO/IAEA/IUPAC Workshop on Method Validation scheduled to be held from 27-29 October 1999 in Budapest, where the Guidelines on In-House Validation would also be considered.
- 50. The Committee welcomed the paper. However, some delegations stressed that the paper did not and should not discourage performing collaborative studies. The Delegation of France informed the Committee that AFNOR VO3-110 containing an intra-laboratory validation protocol had just been revised and would be published and sent to IUPAC.
- 51. The Committee **decided** to request the Netherlands, together with France and the United States, to prepare a paper on the use of information from the proficiency testing studies for the elaboration of characteristics of in-house validated methods for consideration by the Committee at its next session. The Committee **agreed** that when the next draft of the Harmonized Guidelines became available, it

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CX/MAS 98/9, CX/MAS 98/8-Add.1 (recommendations of the Joint FAO/IAEA Expert Consultation on Validation of Analytical Methods for Food Control (December 1997)), CRD 16 (comments from Argentina).

would consider the text to determine if it would be appropriate to recommend it to the Commission for adoption by reference for Codex purposes.

## ENDORSEMENT OF METHODS OF ANALYSIS PROVISIONS IN CODEX STANDARDS (Agenda Item $10)^{\,14}$

52. The report of the *ad hoc* Working Group on Endorsement was presented by its chairperson, Dr. William Horwitz (USA). Ms. Harriet Wallin (Finland) served as rapporteur of the Working Group. The following countries and international organizations participated in the Working Group: Argentina, Australia, Canada, Finland, France, Germany, Hungary, Japan, Republic of Korea, Norway, the United Kingdom, the United States, AOAC International, IDF, ISO and NMKL.

#### **Endorsement of Method of Analysis Provisions**

- 53. The Committee **agreed** to the proposal of the Working Group to revise the endorsement status of two different methods for the determination of potassium in food grade salt that had been previously endorsed together as one Type II method: ESPA/CN-E/104-1994 was endorsed as a Type II method and ESPA/CN-E/103-1994 as a Type III method.
- 54. The Committee did not endorse a number of methods proposed for milk products as either the method had not been demonstrated to be applicable to the product or there could be only one Type I method endorsed for one analyte/product combination.
- 55. The Committee **amended** the methods for determining salt to clarify that the methods determined chloride and the result would be expressed in sodium chloride. The Codex Committee on Processed Fruits and Vegetables would be requested to consider appropriateness of four significant figures for the maximum level for tin in pickles. For some other methods, the Committee recommended to consider the use of more modern methods instead of currently proposed methods.
- 56. A list of methods considered along with their status, assigned Types, and notes containing rationale for non-endorsement and temporary endorsement is attached to this report as Part 1 of Appendix III for approval by the Commission.

#### **Endorsement of Method of Sampling Provisions**

- 57. The Committee considered the sampling provisions of the standards for kimchi, pickles and milk products in the plenary. The Committee **endorsed** those for kimchi and pickles which used the Codex Sampling Plans for Prepackaged Foods, the only document currently available in Codex on sampling and commonly referred to in Codex commodity standards.
- 58. The Committee was informed that IDF Standard 50C provides general instruction for physically obtaining samples while IDF Standards 113A and 136A are statistical sampling plans. The Committee **agreed** to endorse those sampling provisions proposed by the Codex Committee on Milk and Milk Products and to inform the Commission and that Committee of the contradiction in the additional text in the Standard for Cheeses in Brine concerning "cloth or non-absorbent paper".
- 59. In making the above decision the Committee noted that after the adoption of the General Guidelines on Sampling by the Commission as a final text, it might need to review all sampling provisions of the Codex commodity standards. A list of sampling provisions considered by the Committee is attached to this report as Part 2 of Appendix III for approval by the Commission.

CX/MAS 98/9; CRD 2 (Report of the *ad hoc* Working Group on Endorsement of Methods of Analysis Provisions in Codex Standards), CRD 7 (Comments from Hungary), CRD 9 (Comments from South Africa).

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#### **Other Related Matters**

60. During the consideration of the sampling provisions, it was pointed out that information on the selection of sampling plan required in the *Procedural Manual*<sup>15</sup> had not been submitted to this Committee. It was also pointed out that in the case of methods of analysis, no information was generally submitted by the commodity committees despite the *Recommendations for a Checklist of Information Required to Evaluate Methods of Analysis and Sampling for Endorsement*<sup>16</sup> required it. The Committee **agreed** to ask commodity committees to provide information as required by the Checklists to this Committee when they send methods of analysis and sampling to this Committee for endorsement.

- 61. The Committee noted that AOAC 971.20 which had been endorsed as a Type II Codex general method for the determination of copper had only been validated for tea, but not for foods in general. It would consider at its next Session whether this method should be replaced by a method validated for foods in general.
- 62. The Committee **recommended** that commodity committees should select methods from the exiting Codex general methods wherever possible. It also **recommended** that commodity committees should use the SI unit system in the specifications of the standards they develop. The Committee noted that the Codex General Standard for the Labelling of Prepackaged Foods also refers to the use of the SI unit system in relation to net weight and drained weight.
- 63. The Committee **agreed** to establish a new ad hoc Working Group for Endorsement under the Chairship of the United States at its next Session.

#### REPORT OF INTER-AGENCY MEETING ON METHODS OF ANALYSIS (Agenda Item 11) 17

- 64. The report of the 13th Inter-Agency Meeting (IAM)<sup>18</sup> was presented by the Observer from AOAC International. The Committee was informed that the report would be available on the AOAC International's World Wide Web homepage (http://www.aoac.org). An updated *Directory of Organizations Working in the Fields of Standard Methods of Analysis and Laboratory Quality Assurance for the Food Sector*<sup>19</sup> was made available for this Session. The Committee noted that Dr. Roger Wood was elected as chairperson of the 13th Session of the IAM and EURACHEM was accepted as a new member of the IAM.
- 65. The Committee was informed that the IAM noted the recent concerns expressed by several Codex Committees, in particular, the Codex Committee on Pesticide Residues (CCPR) and the Codex Committee on Residues of Veterinary Drugs in Foods (CCRVDF), that it was difficult to find validated methods and information on the validation of methods was not readily available. The Committee was also informed that a general discussion was held on possible publication of performance characteristics in the methods. The IAM agreed to advise that stakeholders<sup>20</sup> of standardized methods of analysis might require the publication of method performance characteristics in the method itself (see para. 34).

<sup>15</sup> Codex Alimentarius Commission Procedural Manual, Tenth Edition, page 65.

<sup>16</sup> Codex Alimentarius, Volume 13, pp 129-134.

<sup>17</sup> CRD 3 (Report of the 13th Inter-Agency Meeting).

AOAC International, Codex Alimentarius Commission, EURACHEM, European Organization for Quality (EOQ), International Association for Cereal Sciences and Technology (ICC), International Atomic Energy Agency (IAEA; Joint FAO/IAEA Division), International Commission for Uniform Methods of Sugar Analysis (ICUMSA), International Dairy Federation (IDF), International Food Law Association (IFLA), International Organization for Standardization (ISO), International Vine and Wine Office (OIV), and Nordic Committee on Food Analysis (NMKL) participated in the IAM.

<sup>19</sup> CRD.

Laboratories, regulators, users of analytical results, etc.

66. The Committee noted that the criteria approach as opposed to prescribing methods of analysis was not appreciated by all IAM members.

- 67. The Committee noted that the IAM considered the need and the definition(s) of "Limit(s)", and presented a report to this Committee. The IAM was of the opinion that if the Codex limit given for a certain matrix was not low enough to come close to the detection limit of a method, there was no need to give limits of determination (see para. 37).
- 68. In relation to the IAM's role and involvement in the quality assurance in the food analysis, the IAM concluded that this issue was progressing in the scientific fora such as the International Harmonization Programme, EURACHEM and others. Regarding the IUPAC/ISO/AOAC Harmonization Programme, the IAM concluded that the harmonization of in-house validation was an issue where the cooperation of all parties involved was required (see paras 49-50). The IAM expressed its wish that more consideration should be given to the use of proficiency testing data in obtaining information on the performance of the methods of analysis used.
- 69. The Committee noted the report of the IAM and appreciated efforts of the IAM in providing technical support to the work of this Committee.

#### OTHER BUSINESS AND FUTURE WORK (Agenda Item 12)

Consideration of the Relationship between the Analytical Result, the Measurement Uncertainty and the Specification in Codex Standards <sup>21</sup>

- 70. The Delegation of the United Kingdom, supported by several delegations, proposed that guidance should be prepared on the interpretation of analytical results in relation to the compliance to the specifications in Codex Standards since there were differences in the treatment of analytical errors in the interpretation of results. The Committee **agreed** to request the United Kingdom, in collaboration with Finland, France, Ireland, Netherlands and the United States, to prepare a paper on this issue for consideration by the Committee at its next Session. It also **agreed** that since the issue involved measurement uncertainty, it would be discussed under the agenda item for measurement uncertainty (see also paras 42-46).
- 71. The Delegation of the United Kingdom noted that it would be of interest for this Committee to follow the progress of matters taken place in other bodies regarding "fitness-for-purpose" of food analysis and sampling.

#### Registration of Laboratories

- 72. Argentina, Brazil, Colombia, Costa Rica, Cuba, Portugal, Spain and Uruguay jointly suggested that the idea of an "International Registration of Laboratories of Non-compulsory and Consultative Nature" be considered for promoting technical exchange in the field of competence of laboratories. It was stated that this specialized laboratories might be considered as potential participants for interlaboratory tests that might be useful in achieving full validation of methods. It was emphasized that it was not easy for developing countries to coordinate inter-laboratory tests and the creation of such a registration system would facilitate full validation of methods.
- 73. The Delegation of the Netherlands informed the Committee that a new version of the *Who's Who in Food Chemistry* had become available and that it includes a number of food analysts.

CRD 18 (proposal from the United Kingdom).

#### Revision of Volume 13

74. The Committee was informed that the publication of a revised version of the *Codex Alimentarius*, Volume 13, was planned in the latter half of the year 1999. The new version would include, in addition to the updated data base of the endorsed methods and other contents of the current version: (1) the adopted harmonized protocols and guidelines by reference; and (2) those methods stated only in Codex standards.

#### Translation of Working Documents and Draft Reports

75. The Delegation of France expressed a serious concern about the late availability of the French version of working documents, and to some extent the Spanish version, which made the preparation for the Session very difficult and requested timely translation of working documents into French and Spanish. The Delegation also requested the translation of the draft reports of future sessions into French and offered to provide a French rapporteur to help the translation process. The Delegation of Spain also offered to provide a Spanish rapporteur for the translation of the draft reports into Spanish. The Chairperson promised to provide the French and Spanish versions of the draft reports at future sessions.

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

76. The committee was informed that its 23rd Session was tentatively scheduled to be held in Budapest in March/April 2000. The exact date and place would be determined between the Host government and Codex Secretariats.

#### ANNEX I

#### **SUMMARY STATUS OF WORK**

Subject	Step	Action by	Document Reference (ALINORM 97/23A)
Proposed Draft General Guidelines on Sampling	3	France Australia, Hungary, Netherlands, United Kingdom, United States, IDF 23rd CCMAS	paras 9-13
Criteria for Evaluating Acceptable Methods of Analysis for Codex Purposes - Guidelines on the Application of the Criteria Approach by the Codex Committee on Methods of Analysis and Sampling	2	United Kingdom Canada, Australia, Finland, France, Germany, Netherlands, Norway, United States Codex Secretariat 23rd CCMAS	Para. 27
Amendments to the Codex Alimentarius Commission Procedural Manual  - Principles for the Establishment of Codex Methods of Analysis and Sampling  - Relations between Commodity Committees and General Committees	1,2,3	23rd CAC Governments 23rd CCMAS	para. 28
Harmonization of Analytical Terminology - "Measurement Limits"	2	United States Finland, France, Spain 23rd CCMAS	Paras 36-40
Harmonization of Reporting of Test Results Corrected for Recovery Factors	2	IUPAC Codex Secretariat Governments 23rd CCMAS	Paras 32-35
Measurement Uncertainty	2	EURACHEM United Kingdom Finland, France, Ireland, Netherlands, United States 23rd CCMAS	para. 37 para. 70
In-House Method Validation	2	Netherlands France, United States IUPAC 23rd CCMAS	paras 47-51
Endorsement of Methods of Analysis and Sampling Provisions in Codex Standards	_22	Commodity Committees Codex Secretariat 23rd CCMAS	

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At various Steps of the Codex Procedure depending on the Steps of the Standards which contain these methods.

#### ALINORM 99/23 APPENDIX I

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#### PROPOSED DRAFT AMENDMENTS TO THE PROCEDURAL MANUAL

(At Steps 1/2/3 of the Procedure)<sup>23</sup>

#### 1. PRINCIPLES FOR THE ESTABLISHMENT OF CODEX METHODS OF ANALYSIS

Addition of a new subsection at the end as follows:

"

#### (C) General Criteria for the Selection of Methods of Analysis using the Criteria Approach

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

## 2. RELATIONS BETWEEN COMMODITY COMMITTEES AND GENERAL COMMITTEES - METHODS OF ANALYSIS AND SAMPLING

Addition of new paragraphs at the end of "Normal Process" section as follows:

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

In addition, the Codex Committee on Methods of Analysis and Sampling will identify numeric values for the criteria for which it would wish such methods to comply."

-

Subject to approval of the Codex Alimentarius Commission as new work.

#### METHODS OF ANALYSIS AND SAMPLING CONSIDERED FOR ENDORSEMENT BY THE COMMITTEE AT THE TWENTY-SECOND SESSION

This Appendix consists of two parts as follows:

- Part 1. Methods of Analysis Provisions of Certain Commodity Standards
- Part 2. Methods of Sampling Provisions of Certain Commodity Standards

#### PART 1 METHODS OF ANALYSIS PROVISIONS OF CERTAIN COMMODITY STANDARDS

#### A. Proposed by Codex Committee on Nutrition and Foods for Special Dietary Uses

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status <sup>24</sup>
Food Grade Salt	Iodine No level specified	ESPA/CN-E/109-1994	Titrimetry using sodium thiosulphate		II	Е
Food Grade Salt	Iodine No level specified	AOAC 925.56	Titrimetry using sodium thiosulphate		III	Е
Food Grade Salt <sup>25</sup>	Potassium [to be filled before publication]	ESPA/CN-E/104-1994	Flame atomic absorption spectrometry	Renumbering of the reference to the method; revision of the previous decision on the Type of the method.	II	Е
Food Grade Salt <sup>25</sup>	Potassium [to be filled before publication]	ESPA/CN-E/103-1994	Titrimetry	Renumbering of the reference to the method; revision of the previous decision on the Type of the method.	III	Е
Guidelines for Nutrition Labelling	Polyunsaturated fat	AOCS Ce 1c-89	Gas liquid chromatography	Previous status, temporarily endorsed.	IV	Е
Guidelines for Nutrition Labelling	Polyunsaturated fat	AOAC 996.06	Gas liquid chromatography	AOAC is requested to clarify whether the method is applicable to the determination of polyunsaturated fat.	II	TE
Guidelines for Nutrition Labelling	Saturated fat	AOCS Ce 1c-89	Gas liquid chromatography	Previous status, temporarily endorsed.	IV	Е

E, endorsed; TE, temporarily endorsed; and NE, not endorsed.

Developed by the Codex Committee on Food Additives and Contaminants.

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status <sup>24</sup>
Guidelines for	Saturated fat	AOAC 996.06	Gas liquid chromatography	IUPAC is requested to submit information	II	Е
Nutrition Labelling				on its available methods for this purpose.		

#### B. Proposed by Codex Committee on Processed Fruits and Vegetables/Codex Coordinating Committee for Asia

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Pickles	Acidity Not specified	AOAC 942.15	Titrimetry		I	Е
Pickles	Acidity Not specified	ISO 750:1981	Titrimetry	This method was not endorsed since there can only be one Type I method for the same provision.		NE
Pickles	Arsenic ≤ 1.0 mg/kg	AOAC 952.13 (Codex general method)	Colorimetry, diethyldithiocarbamate	-	II	Е
Pickles	Arsenic ≤1.0 mg/kg	ISO 6634:1982	Spectrophotometry, silver diethyldithiocarbamate		III	Е
Pickles	Benzoic acid ≤ 250 mg/kg	ISO 5518:1978	Spectrophotometry	The Commodity Committee is requested to review more modern methods such as the liquid chromatographic method IFU 63 (1995) or the gas chromatographic method NMKL 103 (1984)/AOAC 983.16 which has been endorsed as a Type II Codex general method.	IV	TE
Kimchi	Drained weight ≥ 80%	AOAC 968.30	Gravimetry		I	Е
Pickles	Drained Weight Not specified	AOAC 968.30	Gravimetry		I	Е
Pickles	Lead ≤ 1.0 mg/kg	AOAC 972.25 (Codex general method)	Atomic absorption spectrophotometry		II	Е
Pickles	Lead ≤ 1.0 mg/kg	ISO 6633:1984	Flameless atomic absorption spectrophotometry		IV	TE
Kimchi	Mineral impurities ≤ 0.03% m/m	AOAC 971.33	Ashing		I	E
Pickles	Salt Not specified	AOAC 971.27 (Codex general method)	Potentiometry (Determination of chloride, expressed as sodium chloride)		II	Е

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Pickles	Salt Not specified	AOAC 939.10	Volumetry, Gravimetry, Titrimetry (3 methods) (Determination of chloride, expressed as sodium chloride)		III	Е
Kimchi	Salt (sodium chloride) 1.0-4.0% m/m	AOAC 971.27 (Codex general method)	Potentiometry (Determination of chloride, expressed as sodium chloride)		II	Е
Pickles	Sorbates ≤ 1000 mg/kg	ISO 5519:1978	Spectrophotometry	The Commodity Committee is requested to review more modern methods such as the liquid chromatographic method IFU 63 (1995) or the gas chromatographic method NMKL 103 (1984)/AOAC 983.16 which has been endorsed as a Type II Codex general method.	IV	TE
Pickles	Sulphur dioxide ≤ 30 mg/kg	ISO 5522:1981	Titrimetry followed by: gravimetry (high levels) nephelometry (low levels)	The Commodity Committee is requested to review the Optimized Monier-Williams method (AOAC 990.28), which has been endorsed as a Type II Codex general method.		NE
Pickles	Sulphur dioxide ≤ 30 mg/kg	ISO 5523:1981	Colorimetry	See above		NE
Pickles	Tin ≤ 250.0 mg/kg	AOAC 980.19 (Codex general method)	Atomic absorption spectrophotometry	The Commodity Committee is asked to consider whether it is necessary to express the provision using four significant figures.	II	Е
Pickles	Tin ≤ 250.0 mg/kg	ISO 2447:1974		The Commodity Committee is asked to consider whether it is necessary to express the provision using four significant figures.	IV	TE
Kimchi	Total acidity ≤ 1.0% m/m	AOAC 942.15	Tritrimetry	•	I	Е

#### C. Proposed by Codex Committee on Milk and Milk Products

#### 1. Requirements/Specifications in standards (except food additives)

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Whey Powders	Ash ≤ 9.5% (whey powder), ≤ 15.0% (acid whey powder)	IDF Standard 90:1979 (confirmed 1986) ISO 5545:1978	Furnace, 825°C		IV	Е
Edible Casein Products	Ash (including P <sub>2</sub> O <sub>5</sub> ) $\geq$ 7.5% (rennet casein), $\leq$ 2.5% (acid casein)	IDF Standard 90:1979 (confirmed 1986) ISO 5545:1978	Furnace, 825°C		IV	Е
Milkfat Products	Certain antioxidants (use or non-use)	IDF Standard 165:1993	Reversed phase gradient liquid chromatography		II	Е
Milk products	Copper ≤ 5 mg/kg (whey powders, edible casein products) ≤ 0.05 mg/kg (butter, milkfat products)	AOAC 971.20 (Codex general method)	Atomic absorption spectrophotometry	The Committee was informed that the method had been validated for tea only.		NE
Milk Products	Copper ≤ 5 mg/kg (whey powders, edible casein products)	AOAC 985.35	Atomic absorption spectrophotometry	This method was endorsed in place of AOAC 971.20 (see above); the provisions for copper in milk products was separated into two separate entries on the basis of the applicability of the method.	II	Е
Milk Products	Copper ≤ 5 mg/kg (whey powders, edible casein products)	IDF Standard 76A:1980 ISO 5738:1980 AOAC 960.40 (Codex general method)	Photometry, diethyldithiocarbamate		III	Е
Milk Products	Copper ≤ 0.05 mg/kg (butter, milkfat products)	IDF Standard 76A:1980 ISO 5738:1980 AOAC 960.40 (Codex general method)	Photometry, diethyldithiocarbamate	The Committee was not convinced of the applicability of the method to high fat products. The Commodity Committee is requested to review the method in this respect as well as to consider the applicability of method AOAC 990.05, IUPAC Method Pure and Applied Chem., 60, No6 (atomic absorption spectrophotometry, graphite furnace).		NE

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Cheeses in Brine	Dry matter (for composition)	IDF Standard 4A:1982 ISO 5534:1985	Gravimetry, drying at 102°C	The Commodity Committee is asked to forward its recommendation as to which of the two Type I methods it prefers since only one Type I method can be endorsed for the one analyte/product combination.		NE
Cheeses in Brine	Dry matter (for composition)	AOAC 926.08	Gravimetry, vacuum oven	See above.		NE
Whey Cheese	Dry matter (for denomination)	IDF Standard 58:1970 (confirmed 1993) ISO 2920:1974	Gravimetry, drying at 88±2°C	The Commodity Committee may wish to consider if the provision can be handled by other methods.	IV	Е
Edible Casein Products	Free acid ≤ 0.27 ml-0.1 N NaOH/g	IDF Standard 91:1979 (confirmed 1986) ISO 5547:1978	Titrimetry, aqueous extract		IV	Е
Milkfat Products	Free fatty acids (expressed as oleic acid) ≤ 0.3% (anhydrous milkfat, anhydrous butteroil) ≤ 0.4% (milkfat, butteroil, ghee)	IDF Standard 6B:1989 ISO 1740:1991 AOAC 969.17	Titrimetry	For consistency, the method is endorsed as Type I since a conversion factor is included in the method.	I	Е
Milk Products	Iron ≤ 20 mg/kg (spray dried whey powder, edible caseinate products except roller dried caseinates), ≤ 50 mg/kg (roller dried whey powder & caseinates) ≤ 2.0 mg/kg (butter) ≤ 0.2 mg/kg (milkfat products)	NMKL 139 (1991) (Codex general method)	Atomic absorption spectrophotometry	The level ≤0.2 mg/kg was added to this provision since it was presumed that its omission was a typographical error (see below).	II	Е
Milk Products	Iron ≤ 20 mg/kg (spray dried whey powder, edible caseinate products except roller dried caseinates), ≤ 50 mg/kg (roller dried whey powder & caseinates) ≤ 2.0 mg/kg (butter) ≤ 0.2 mg/kg (milkfat products)	IDF Standard 103A:1986 ISO 6732:1985	Photometry, bathophenanthroline		IV	Е
Edible Casein Products	Lactose ≤ 1.0%	IDF Standard 106:1982 ISO 5548:1980	Photometry, phenol and H <sub>2</sub> SO <sub>4</sub>		IV	Е

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Whey Powders	Lactose (expressed as anhydrous lactose)	IDF Standard 79B:1991	Enzymatic method; glucose moiety (method A),	The Commodity Committee is requested to submit information as to		NE
	≥ 61.0%	ISO/DIS, Part I and Part II	galactose moiety (method B)	which method (A or B) it prefers.		
Butter	Lead	AOAC 972.25 (Codex	Atomic absorption		II	Е
	≤ 0.05 mg/kg	general method)	spectrophotometry			
Edible Casein	Lead	AOAC 972.25 (Codex	Atomic absorption		II	E
Products	≤ 1 mg/kg	general method)	spectrophotometry			
Edible Casein	Lead	IDF Standard	Spectrometry, 1,5-		III	E
Products	≤ 1 mg/kg	133A:1992	diphenylthiocarbazone			
Edible Casein	Lead	AOAC 982.23 (Codex	Anodic Stripping Voltammetry		III	Е
Products	≤ 1 mg/kg	general method)				
Edible Casein	Lead	NMKL 139 (1991)	Atomic absorption		III	Е
Products	≤ 1 mg/kg	(Codex general method)	spectrophotometry			
Whey Powders	Lead	AOAC 972.25 (Codex	Atomic absorption		II	Е
·	≤ 1 mg/kg	general method)	spectrophotometry			
Butter	Milk solids-not-fat	IDF Standard 80:1977	Gravimetry		I	Е
	≤ 2%	ISO 3727:1977	•			
		AOAC 920.116				
Butter	Milkfat	IDF Standard 80:1977	Gravimetry		I	E
	≥ 80%	ISO 3727:1977				
		AOAC 938.06				
Cheese	Milkfat	IDF Standard 5B:1986	Gravimetry (Schmid-		I	E
	(specified in individual standards)	ISO 1735:1987	Bondzynski-Ratslaff)			
	2 1111 2	AOAC 933.05				
Edible Casein	Milkfat	IDF Standard	Gravimetry (Schmid-		I	E
Products	≤ 2.0%	127A:1988 ISO 5543:1986	Bondzynski-Ratslaff)			
Evaporated	Milkfat	IDF Standard	Gravimetry (Röse-Gottlieb)		I	Е
Milks	$\geq$ 7.5% (evaporated milk), $\leq$ 1.0%	13C:1987	•			
	(evaporated skimmed milk), >1.0% &	ISO 1737:1985				
	<7.5% (evaporated partly skimmed milk), ≥15.0% (evaporated high-fat milk)	AOAC 945.48G				

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Evaporated Milks	Milkfat ≥7.5% (evaporated milk), ≤ 1.0% (evaporated skimmed milk), >1.0% & <7.5% (evaporated partly skimmed milk), ≥15.0% (evaporated high-fat milk)	AOAC 920.115F	Gravimetry (Röse-Gottlieb)	This method was not endorsed since there can be only one Type I method; in addition the Committee was informed that the method had not been shown to be applicable to the commodity in question.		NE
Milk Powders and Cream Powders	Milkfat ≥ 42% (cream powder), ≥ 26% & <42% (whole milk powder), >1.5% & <26% (partly skimmed milk powder), ≤ 1.5% (skimmed milk powder)	IDF Standard 9C:1987 ISO 1736:1985 AOAC 932.06	Gravimetry (Röse-Gottlieb)		I	Е
Milk Powders and Cream Powders	Milkfat ≥ 42% (cream powder), ≥ 26% & <42% (whole milk powder), >1.5% & <26% (partly skimmed milk powder), ≤ 1.5% (skimmed milk powder)	AOAC 920.115F	Gravimetry (Röse-Gottlieb)	This method was not endorsed since there can be only one Type I method; in addition the Committee was informed that the method had not been shown to be applicable to the commodity in question.		NE
Milk Powders and Cream Powders	Milkfat ≥ 42% (cream powder), ≥ 26% & <42% (whole milk powder), >1.5% & <26% (partly skimmed milk powder), ≤ 1.5% (skimmed milk powder)	AOAC 945.48G	Gravimetry (Röse-Gottlieb)	This method was not endorsed since there can be only one Type I method; in addition the Committee was informed that the method had not been shown to be applicable to the commodity in question.		NE
Milk Products (for products not completely soluble in ammonia)	Milkfat	IDF Standard 126A:1988 ISO 8262-3:1987	Gravimetry (Weibull-Berntrop)	•	I	Е
Milkfat Products	Milkfat ≥ 99.8% (anhydrous milkfat, anhydrous butteroil) ≥ 99.6% (milkfat, butter oil, ghee)	IDF Standard 24:1964	Gravimetry (calculation for solids-not-fat and water content)		IV	Е

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Sweetened Condensed Milks	Milkfat ≥ 8.0% (sweetened condensed milk), ≤ 1.0% (sweetened condensed skimmed milk), >1.0% & <8.0% (sweetened condensed partly skimmed milk), >16.0% (sweetened condensed high-fat milk)	IDF Standard 13C:1987 ISO 1737:1985 AOAC 920.115F	Gravimetry (Röse-Gottlieb)		I	E
Sweetened Condensed Milks	Milkfat ≥ 8.0% (sweetened condensed milk), ≤ 1.0% (sweetened condensed skimmed milk), >1.0% & <8.0% (sweetened condensed partly skimmed milk), >16.0% (sweetened condensed high-fat milk)	AOAC 945.48G	Gravimetry (Röse-Gottlieb)	This method was not endorsed since there can be only one Type I method; in addition the Committee was informed that the method had not been shown to be applicable to the commodity in question.		NE
Whey Powders	Milkfat ≤2%	IDF Standard 9C:1987 ISO 1736:1985 AOAC 932.06	Gravimetry (Röse-Gottlieb)		Ι	Е
Whey Cheese	Milkfat (dry basis) ≥ 33% (creamed whey cheese), ≥ 10% & <33% (whey cheese), <10% (skimmed whey cheese)	IDF Standard 59A:1986 ISO 1854:1987 AOAC 974.09	Gravimetry (Röse-Gottlieb)		I	E
Cheeses in Brine	Milkfat in dry matter ≥ 40% (soft, semi-hard)	IDF Standard 5B:1986 ISO 1735:1987 AOAC 933.05	Gravimetry (Schmid- Bondzynski-Ratslaff)		I	Е
Cheese	Moisture (specified in individual standards)	AOAC 926.08	Gravimetry, vacuum oven	The Commodity Committee is asked to forward its recommendation as to which of the two Type I methods it prefers since only one Type I method can be endorsed for the one analyte/product combination.		NE
Cheese	Moisture (specified in individual standards)	IDF Standard 4A:1982 ISO 5534:1985	Gravimetry, drying at 102°C	This method was added for consistency (see above).		NE
Edible Casein Products	Moisture ≤12% (rennet casein & acid casein), ≤8% (caseinates)	IDF Standard 78C:1991 ISO 5550:1978	Gravimetry, drying at 102°C		Ι	Е

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Whey Powders	Moisture, "Free" ≤5.0% (whey powder), ≤4.5% (acid whey powder)	IDF Standard 58:1970 (confirmed 1993) ISO 2920:1974	Gravimetry, drying at 88±2°C		IV	Е
Milkfat Products (Anhydrous Milkfat)	Peroxide value ≤0.3 milliequivalents of oxygen/kg fat	AOAC 965.33	Titrimetry		I	Е
Milkfat Products	Peroxide value (expressed as milliequivalents of oxygen/kg fat) ≤0.6 (milkfat, butteroil, ghee) ≤0.3 (anhydrous milkfat, anhydrous butteroil)	IDF Standard 74A:1991 ISO 3976:1977	Photometry, FeCl <sub>3</sub> /NH <sub>4</sub> CNS	The Commodity Committee is requested to consider whether AOAC 965.33 is applicable to the determination of peroxide values in these milkfat products (in addition to anhydrous milkfat).		NE
Edible Casein Products	PH ≤7.5 (caseinates)	IDF Standard 115A:1989 ISO 5546:1979	Electrometry		IV	Е
Evaporated Milks	Protein (in milk solids-not-fat) ≥ 34%	AOAC 945.48H	Kjeldahl, titrimetry		I	Е
Evaporated Milks	Protein (in milk solids-not-fat) ≥ 34%	IDF Standard 20B:1993 AOAC 991.20-23	Kjeldahl, titrimetry,	This method was not endorsed since there can be only one Type I method; in addition the Committee was informed that the method had not been shown to be applicable to the commodity in question.		NE
Milk Powders and Cream Powders	Protein (in milk solids-not-fat) ≥ 34%	IDF Standard 20B:1993 AOAC 991.20-23	Kjeldahl, titrimetry		Ι	Е
Sweetened Condensed Milks	Protein (in milk solids-not-fat) ≥ 34%	AOAC 920.115G	Kjeldahl, titrimetry		I	Е
Sweetened Condensed Milks	Protein (in milk solids-not-fat) ≥ 34%	IDF Standard 20B:1993 AOAC 991.20-23	Kjeldahl, titrimetry	This method was not endorsed since there can be only one Type I method; in addition the Committee was informed that the method had not been shown to be applicable to the commodity in question.		NE

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Edible Casein Products	Protein (Total N x 6.38 in dry matter) $\geq$ 84% (rennet casein), $\geq$ 90% (acid casein), $\geq$ 88% (caseinates)	IDF Standard 92:1979 (confirmed 1986) ISO 5549:1978	Kjeldahl, titrimetry		IV	Е
Whey Powders	Protein (Total N x 6.38) $\geq 11\%$ (whey powder), $\geq 10\%$ (acid whey powder)	IDF Standard 92:1979 (confirmed 1986) ISO 5549:1978	Kjeldahl, titrimetry		IV	E
Butter	Salt (for labelling purposes)	IDF Standard 12B:1988 ISO 1738:1997 AOAC 960.29	Titrimetry (Determination of chloride, expressed as sodium chloride)		II	E
Butter	Salt (for labelling purposes)	IDF Standard 179:1997 AOAC 971.27 (Codex general method)	Potentiometry (Determination of chloride, expressed as sodium chloride)	This method was added by the Committee. The Commodity Committee is asked to consider this Codex general method as an alternative method.	III	Е
Milk Powders and Cream Powders	Scorched Particles Max disc B	IDF Standard 107A:1995 ISO 5739:1983	Visual comparison with standard discs, after filtration		IV	Е
Edible Casein Products	Sediment (scorched particles)(in 25 g) ≤15 mg (rennet casein), ≤22.5 mg (acid casein, spray dried caseinates), ≤81.5 mg (roller dried caseinates)	IDF Standard 107A:1995 ISO 5739:1983	Visual comparison with standard disks, after filtration		IV	Е
Cheese	Solids (specified in individual standards)	IDF Standard 4A:1982 ISO 5534:1985	Gravimetry, drying at 102°C	The Commodity Committee is asked to forward its recommendation as to which of the two Type I methods it prefers since two methods for the same provision cannot be endorsed.		NE
Cheese	Solid (specified in individual standards)	AOAC 926.08	Gravimetry, vacuum oven	See above.		NE
Evaporated Milks	Solids ≥ 25% (evaporated milk), ≥ 20% (evaporated skimmed milk, evaporated partly skimmed milk)	IDF Standard 21B:1987 ISO 6731:1989 AOAC 925.23A	Gravimetry, drying at 98-100°C	The reference AOAC 945.48D was deleted as being not applicable.	I	Е

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Evaporated Milks	Solids ≥ 25% (evaporated milk), ≥ 20% (evaporated skimmed milk, evaporated partly skimmed milk)	AOAC 920.107+925.23A	Gravimetry, drying at 98-100°C	This method was not endorsed since there can be only one Type I method; in addition the Committee was informed that the method had not been shown to be applicable to the commodity in question.		NE
Sweetened Condensed Milks	Solids ≥ 28% (sweetened condensed milk), ≥ 24% (sweetened condensed skimmed milk, sweetened condensed partly skimmed milk	IDF Standard 15B:1991 ISO 6734:1989	Gravimetry, drying at 102°C	The Commodity Committee is asked to forward its recommendation as to which of the two Type I methods it prefers since two methods for the same provision cannot be endorsed.		NE
Sweetened Condensed Milks	Solids ≥ 28% (sweetened condensed milk), ≥ 24% (sweetened condensed skimmed milk, sweetened condensed partly skimmed milk	AOAC 920.115D	Gravimetry, vacuum oven	See above.		NE
Milk Powders and Cream Powders	Solubility ≤1.0 ml	IDF Standard 129A:1988 ISO 8156:1987	Centrifugation		I	Е
Milk Powders and Cream Powders	Titratable acidity ≤18.0 ml-0.1N NaOH/10 g-solids-no- fat	IDF Standard 86:1981	Titrimetry, titration to pH 8.4		I	Е
Milk Powders and Cream Powders	Titratable acidity ≤18.0 ml-0.1N NaOH/10 g-solids-no- fat	IDF Standard 81:1981	Titrimetry	This method using phenolphthalein was not endorsed as it was considered that the above method covered the provision and there can be only one Type I method for a provision.		NE
Butter	Vegetable fat (free from vegetable fat)	IDF Standard 32:1965 ISO 3595:1976 AOAC 955.34A	Phytosteryl acetate test		III	Е
Butter	Vegetable fat (free from vegetable fat)	IDF Standard 54:1970 ISO 3594:1976 AOAC 970.50A	Gas liquid chromatography		II	Е
Milkfat Products	Vegetable fat (free from vegetable fat)	IDF Standard 32:1965 ISO 3595:1976 AOAC 955.34A	Phytosteryl acetate test		III	Е

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Milkfat	Vegetable fat (sterols)	IDF Standard 54:1970	Gas liquid chromatography		II	Е
Products	(free from vegetable fat)	ISO 3594:1976				
		AOAC 970.50A				
Butter	Water	IDF Standard 80:1977	Gravimetry		I	Е
	≤16%	ISO 3727:1977				
		AOAC 920.116				
Milk Powders	Water	IDF Standard	Gravimetry, drying at 102°C		IV	Е
and Cream	≤5%	26A:1993				
Powders						
Milkfat	Water	IDF Standard	Titrimetry (Karl Fischer)		II	Е
Products	≤0.1% (anhydrous milkfat, anhydrous	23A:1988				
	butteroil)					

## 2. Methods established for food additives

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Cheese and Processed Cheese Products	Citric acid	IDF Standard 34C:1992	Enzymatic		II	Е
Cheese and Processed Cheese Products	Citric acid	AOAC 976.15 ISO 2963:1997	Photometry		III	Е
Cheese (and cheese rind)	Natamycin	IDF Standard 140A:1992 ISO 9233:1991	Molecular absorption spectrometry & HPLC after extraction		П	Е

# D. Proposed by Codex Committee on Fish and Fishery Products

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Quick Frozen Fish Sticks (fish fingers) and Fish Portions-Breaded and in Batter(except for certain fish species with soft flesh)	Proportion of fish fillet and minced fish Not specified	See Annex 2 to CX/MAS 98/9	Gravimetry	The Commodity Committee is asked to review the collaborative study data, which was available late at this session.		NE

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Quick Frozen Fish Sticks (fish fingers) and Fish Portions-Breaded and in Batter(for certain fish species with soft flesh, such as hakes from the Southern Hemisphere)	Proportion of fish fillet and minced fish Not specified	See Annex 3 to CX/MAS 98/9	Gravimetry		IV	Е
Quick Frozen Fish Sticks (fish fingers) and Fish Portions-Breaded and in Batter	Proportion of fish flesh in fish sticks (fish core) Not specified	AOAC 996.15 (with an adjustment factor of 2% for raw breaded and batter-dipped products; 4% for precooked products)	Gravimetry	AOAC 996.15 is a modified method of AOAC 971.13 which was endorsed previously. The matter is referred back to the Commodity Committee for further considerations regarding the application of adjustment factors.	I	TE
Salted Fish of the <i>Gadidae</i> Family	Salt No level specified	See Annex 4 to CX/MAS 98/9	Titrimetry (Mohr)	Endorsement postponed awaiting collaborative study data.		NE

# E. Proposed by Codex Committee on Sugars

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Sugars (fructose)	D-Glucose ≤0.5% m/m	ISO 10504:1998	Liquid chromatography, refractive index detection		II	Е
Sugars (fructose)	D-Fructose =>98% m/m	ISO 10504:1998	Liquid chromatography, refractive index detection		II	Е
Sugars (white sugar, plantation or mill white sugar, soft white sugar, soft brown sugar, powdered sugar, powdered dextrose, raw cane sugar)	Sulphur dioxide ≤70 mg/kg	ICUMSA GS 2/3-35 (1998) NMKL 135 (1990) EN 1988-2 (1998)	Enzymatic method		II	E
Sugars (dextrose anhydrous, dextrose monohydrate, glucose syrup, dried glucose syrup, fructose)	Sulphur dioxide ≤400 mg/kg	ISO 5379:1983	Acidimetry and nephelometry		IV	Е

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Sugars (powdered sugar)	Anticaking agents ≤1.5% m/m	ICUMSA (1994) GS 3-21 to be amended to incorporate a method for the determination of starch to meet the requirements of the standard		The required amendments had not yet been undertaken.		NE
Sugars	Arsenic Free (raw cane sugar) ≤1 mg/kg (others)	AOAC 952.13 (Codex general method)	Colorimetry, diethyldithiocarbamates	This method was added and endorsed by the Committee since it is a Codex general method.	II	Е
Sugars	Arsenic Free (raw cane sugar) ≤1 mg/kg (others)	ICUMSA GS 2/3-25	Colorimetry (diethyldithiocarbamates)		IV	Е
Sugars	Lead Free (raw cane sugar) ≤0.5 mg/kg (less refined sugars) ≤0.1 mg/kg (refined sugar products)	AOAC 997.15	Atomic Absorption Spectrometry (graphite furnace)		II	Е
Sugars (dextrose anhydrous, dextrose monohydrate, dried glucose syrup, glucose syrup, powdered dextrose, lactose)	Sulphated ash ≤0.25 % m/m on a dry basis	ISO 5809:1982	Single sulphonation		I	Е
Sugars (fructose)	Conductivity ash ≤0.1 % m/m	ICUMSA GS 1/3/4/7/8-13 (1994)	Conductimetry	It is recommended that the method be replaced by method ICUMSA GS 2/3-17 (1994).		NE
Sugars (soft sugars, brown sugar)	Invert sugar (applicable at >10% m/m levels)	ICUMSA GS 4/3-3 (1994)	Titrimetry (Lane & Eynon)		I	Е
Sugars (soft white sugar)	Colour ≤60 ICUMSA units	ICUMSA GS 2/3-9 (1994)	Photometry		I	Е
Honey	Sugars added: for sugar profile	AOAC 977.20	Liquid chromatography		II	Е
Honey	Sugars added: detection of high fructose syrup, corn syrup.	AOAC 979.22	Thin layer chromatography		II	Е

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Honey	Sugars added: detection AOAC 978.17 of corn and cane sugar products.		Carbon isotope ratio mass spectrometry		I	Е

#### PART 2 METHODS OF SAMPLING PROVISIONS OF CERTAIN COMMODITY STANDARDS

## A. Proposed by Codex Committee on Processed Fruits and Vegetables/Codex Coordinating Committee for Asia

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Status
Kimchi	Sampling	Codex Sampling Plans for Prepackaged Foods (AQL 6.5)			Е
Pickles	Sampling	Codex Sampling Plans for Prepackaged Foods (AQL 6.5)			Е

# B. Proposed by Codex Committee on Milk and Milk Products

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Status
Butter	Sampling	IDF Standard 50C:1995 ISO 707:1997 AOAC 968.12	General instructions for obtaining a sample from a bulk		Е
Cheese	Sampling	IDF Standard 50C:1995 ISO 707:1997 AOAC 968.12	General instructions for obtaining a sample from a bulk		Е
Cheeses in Brine	Sampling	IDF Standard 50C:1995 ISO 707:1997 AOAC 968.12 A representative piece of cheese is placed on a cloth or on a sheet of non-absorbent paper for 5 to 10 min. A slice of 2-3 cm is cut off and sent to the laboratory in a sealed insulated box for analysis.	General instructions for obtaining a sample from a bulk	The matter is referred to the CAC and the Commodity Committee for clarification as to whether the text in the standard regarding sampling contains contradictions (use of cloth or non-absorbent paper are given as alternatives).	Е
Edible Casein Products	Sampling	IDF Standard 50C:1995 ISO 707:1997 AOAC 968.12	General instructions for obtaining a sample from a bulk		Е

COMMODITY	PROVISION	METHOD	PRINCIPLE Note	Status
Evaporated Milks	Sampling	IDF Standard 50C:1995	General instructions for obtaining	E
		ISO 707:1997	a sample from a bulk	
		AOAC 968.12	-	
Milk Powders and	Sampling	IDF Standard 50C:1995	General instructions for obtaining	Е
Cream Powders		ISO 707:1997	a sample from a bulk	
		AOAC 968.12		
Milk Products	Sampling	IDF Standard 50C:1995	General instructions for obtaining	Е
		ISO 707:1997	a sample from a bulk	
		AOAC 968.12		
Milk Products	Sampling	IDF Standard 113A:1990	Inspection by attributes	E
		ISO 5538:1987		
Milk Products	Sampling	IDF Standard 136A:1992	Inspection by variables	Е
		ISO 8197:1988		
Milkfat Products	Sampling	IDF Standard 50C:1995	General instructions for obtaining	Е
		ISO 707:1997	a sample from a bulk	
		AOAC 968.12		
Sweetened	Sampling	IDF Standard 50C:1995	General instructions for obtaining	Е
Condensed Milks		ISO 707:1997	a sample from a bulk	
		AOAC 968.12		
Whey Cheese	Sampling	IDF Standard 50C:1995	General instructions for obtaining	Е
		ISO 707:1997	a sample from a bulk	
		AOAC 968.12	•	
Whey Powders	Sampling	IDF Standard 113A:1990	Inspection by attributes	Е
•		ISO 5538:1987		
Whey Powders	Sampling	IDF Standard 50C:1995	General instructions for obtaining	E
•		ISO 707:1997	a sample from a bulk	
		AOAC 968.12	•	

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Status
Milk Products	Sampling of milk from Bulk Tanks	AOAC 970.26		The Committee noted that this method has not been included in any of Codex standards but included in the list of methods agreed by the Codex Committee on Milk and Milk Products <sup>26</sup> and that there is no Codex standard for milk. However, as milk is used as a raw material for many standardized products a sampling method for milk may be needed.	Е

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