# codex alimentarius commission

FOOD AND AGRICULTURE **ORGANIZATION** OF THE UNITED NATIONS

WORLD HEALTH ORGANIZATION

JOINT OFFICE:

Via delle Terme di Caracalla 00100 ROME: Tel. 57971 Telex: 610181 FAOI. Cables Foodagri Facsimile: 6799563

ALINORM 89/23

#### JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX ALIMENTARIUS COMMISSION Eighteenth Session Geneva, 3-12 July 1989

REPORT OF THE SIXTEENTH SESSION OF THE CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

Budapest, 14-19 November 1988

Includes also the Report of the Seventh Inter-Agency Meeting Budapest, 10-11 November 1988

NOTE: This document incorporates Codex CL 1989/3-MAS

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CL 1989/3-MAS January 1989

TO:

- Codex Contact Points

- Participants at the 16th Session of the Codex Committee

on Methods of Analysis and Sampling
- Interested International Organizations

FROM:

Chief, Joint FAO/WHO Food Standards Programme, FAO,

Via delle Terme di Caracalla, 00100 Rome, Italy

SUBJECT:

Distribution of the Report of the Sixteenth Session

of the Codex Committee on Methods of Analysis and Sampling (CCMAS)

The report of the Sixteenth Session of the above Committee (ALINORM 89/23) will be considered by the Eighteenth Session of the Codex Alimentarius Commission (Geneva, 3-14 July 1989).

#### PART A - MATTERS OF INTEREST TO THE COMMISSION

#### (1) Guidelines on Sampling

The Commission has been requested to give consideration to the development by the CCMAS, with input from Codex Committees, of a single advisory document on sampling (paras. 13-23 and Appendix II, ALINORM 89/23).

#### (2) Sampling for Net Contents

The OIML sampling plans for net weight have been referred to the Commission for its agreement that the OIML sampling plans be further developed by the CCMAS in accordance with an appropriate procedure (paras. 24-29 and Appendix V, ALINORM 89/23).

#### (3) Sampling Plan for Food Grade Salt

The sampling plan for food grade salt referred to the CCMAS by the Codex Committee on Food Additives and Contaminants has been endorsed and referred to the Commission for adoption and inclusion in the Codex Standards for Food Grade Salt (CODEX STAN. 150-1985) (paras. 31-34 and Appendix III, ALINORM 89/23).

#### (4) Codex General Methods of Analysis for Contaminants

The CCMAS has initiated review of the Codex general methods for analysis for contaminants in foods (paras. 70-71 and Appendix VI, ALINORM 89/23).

#### PART B - MATTERS OF INTEREST TO GOVERNMENTS

(1) Codex Guidelines on Sampling

Action is pending decison by the Commission and the preparation of a first draft of the Guidelines (see Part A (1) above).

(2) Sampling for Net Contents

Following decision by the Commission, Governments will be advised of action to be taken on the OIML Sampling Plans (see para 29 and App. V, ALINORM 89/23).

(3) Guidelines for Classification of Codex Methods of Analysis and the concept of "simple" methods of analysis (see paras. 54-57 and ALINORM 89/23)

Governments and interested International Organizations are requested to send their comments on the U.K. paper given in Appendix VI to ALINORM 89/23 to Mr. R.S. Kirk, Laboratory of the Government Chemist, Queens Road, Teddington, Middlesex TWII OLY, United Kingdom no later than the end of 1989, with a copy to the Chief, Joint FAO/WHO Food Standards Programme, FAO, Via delle Terme di Caracalla, 00100 Rome, Italy.

(4) Review of Codex General Methods of Analysis for Contaminants (see paras. 70-71, ALINORM 89/23)

Governments and interested International Organizations are requested to send their comments and information on the Codex general methods included in Appendix VII, ALINORM 89/23 to the Chief, Joint FAO/WHO Food Standards Programme, FAO, 00100 Rome, Italy not later than the end of 1989, with a copy to Mr. I. Oláh, Head of Department, Hungarian Office for Standardization, H-1450 Budapest 9, Pf. 24, Hungary.

#### SUMMARY AND CONCLUSIONS

The Sixteenth Session of the Codex Committee on Methods of Analysis and Sampling reached the following conclusions during its deliberations:

- (1) A single comprehensive document on <u>sampling</u> should be developed (paras. 13-23, Appendix II).
- (2) The OILM sampling plans for net contents should be sent to Governments for comments and be reconsidered by the CCMAS (paras. 24-29, Appendix V).
- (3) Considered endorsement of sampling plans for food grade salt, other Codex standards and environmental contaminants (paras. 30-44).
- (4) Agreed that the <u>needs of Codex for methods of analysis</u> should be studied and priorities determined (paras. 49, 72-73).
- (5) Decided to develop, with the assistance of AOAC, a general method of ashing (para. 51).
- (6) Agreed to develop guidelines for the classification of Codex methods of analysis (as Types I, II etc., paras. 54-57, Appendix VI).
- (7) Referred the question of defining "limit of determination" or "limit of detection" to IUPAC (paras. 58-62).
- (8) Referred the question of "simple" methods to the interested Codex Committees for consideration (paras. 63-66).
- (9) Considered the endorsement of methods of analysis in various draft Codex standards (paras. 67-69, Appendix IV).
- (10) Decided to review, on the basis of Government comments, the Codex General methods for contaminants (paras. 70-71, Appendix VII).
- (11) Considered the report of the Inter-Agency Meeting (IAM) (paras 45-53, Appendix VIII).
- (12) Defined its future work programme (paras. 74-76).

#### TABLE OF CONTENTS

		Paragraph
ADOPTION OF THE A APPOINTMENT OF R MATTERS OF INTER NATURE AND PURPO GUIDELINES ON SAI REGARD TO SAMP SAMPLING FOR NET ENDORSEMENT OF M  - Sampling - Sampling - Unshelled - Vegetable wheat glu - Standards Codex Coo - Quick fro	AGENDA APPORTEURS EST TO THE COMMITTEE SE OF CODEX METHODS OF SAMPLING MPLING - REVIEW OF CODEX STANDARDS WITH LING CONTENTS ETHODS OF SAMPLING IN CODEX STANDARDS  plans for food grade salt plans included in Draft Codex Standards pistachio nuts and dates protein products, soy protein products, ten and draft standards being elaborated by the rdinating Committee for Africa zen fish fillets and squid, dried shark fins	1 - 4 5 - 6 7 8 - 12 13 - 17 18 - 23 24 - 29 30 - 44 31 - 34 35 36 37
durum whe whole mai - Sampling - Soy bean - Environme	ndards for pulses, sorghum grains and flour, at flour and semolina, wheat flour, maize, ze meal, degermed maize meal and maize grits for aflatoxins in cereals, pulses and legumes oil	41 42 43 - 44
CLASSIFICATION OF LIMIT OF DETERMING SELECTION OF "SI ENDORSEMENT OF METHODS OF ANALY FUTURE WORK OTHER BUSINESS TIME AND PLACE OF COMMETTER SERVICES OF TIME AND PLACE OF COMMETTER SERVICES	VENTH INTER-AGENCY MEETING (IAM)  OF CODEX METHODS OF ANALYSIS  NATION  MPLE" METHODS OF ANALYSIS  ETHODS OF ANALYSIS IN DRAFT STANDARDS  OF ANALYSIS FOR CONTAMINANTS  SIS REQUIRED BY CODEX  OF NEXT SESSION  OF WORK	54 - 57 58 - 62 63 - 66 67 - 69 70 - 71 72 - 73 74 - 76 77 - 81 82
APPENDICES		Page
Appendix I Appendix II	List of Participants	
Appendix III	Method for Sampling of Food Grade Salt for Compositional Criteria	
Appendix IV Appendix V	Report of an ad hoc Working Group on Endorsement of Methods of Analysis	
Appendix VI Appendix VII	in Packages	44 - 48 49 - 52
Appendix VIII	Metallic Contaminants in Food	

#### INTRODUCTION

- 1. The Codex Committee on Methods of Analysis and Sampling held its sixteenth session from 14 to 19 November 1988 in Budapest, by courtesy of the Government of Hungary. The Session was opened by Dr. K. Sütő, President of the Hungarian National Codex Committee, Vice-President of the Hungarian Office for Standardization and Member of Parliament, who welcomed the participants on behalf of the Hungarian National FAO Committee and the Hungarian National Codex Committee. He informed the Committee that, because of illness, Professor R. Lásztity could not chair the present session. Professor P. Biacs had been appointed by the Hungarian Government to act as Chairman of the Sixteenth Session of the Committee. Dr. Sütő expressed the opinion that the work carried out by the Codex Alimentarius Commission over the last 25 years proved that the founders had been correct when they decided to embark on a programme of standardization of food products at the international level. This was also evidenced by the growing membership of the Commission which had reached over 130 member states. The number of international organizations participating in the work of the Commission had also increased. Work in the field of analysis and sampling leading to internationally accepted methods to check compliance with the Codex Standards was essential.
- 2. The Committee expressed its regret concerning Professor Lásztity's illness and wished him speedy recovery.
- 3. The Committee was chaired by Dr. P. Biacs, Professor at the University of Horticulture and Food Industry, Member of Parliament, and General Director of the Central Food Research Institute, Budapest.
- 4. The Session was attended by delegates from 26 countries and observers from 6 international organizations. The list of participants, including the Hungarian Technical Secretariat and officers from the Joint FAO/WHO Food Standards Programme, is attached as Appendix I to this report.

#### ADOPTION OF THE AGENDA (Agenda Item 2)

- 5. The Committee adopted the provisional agenda without any change. It agreed, however, to discuss the methods of analysis and sampling for salt before the end of the third day of the Session.
- 6. In order to ensure that all delegates had an opportunity to study the working papers, the Committee agreed that the afternoon of the first day of the Session be dedicated only to meetings of working groups. The Committee decided to set up a working group on analysis to consider the working documents and other relevant material required for the endorsement of methods of analysis included in Codex standards. The following delegations indicated their interest to participate in the Working Group: Australia, Austria, Canada, China, Cuba, Czechoslovakia, Denmark, Federal Republic of Germany, Finland, Hungary, Netherlands, Norway, Poland, Spain, Sweden, Switzerland, UK and USA, and observers from ECSS, IFG, ISO and OIV. The Committee also decided to set up a working group on sampling to consider especially the question of the purpose and status of Codex sampling procedures and to prepare suggestions for consideration, in plenary session, on how work on sampling within Codex should proceed. A number of delegations indicated their interest to participate in discussions on sampling. Other delegations were also invited to participate. It was understood that the working groups would choose their Chairmen and Rapporteurs.

#### APPOINIMENT OF RAPPORTEURS (Agenda Item 3)

7. The Committee appointed the Delegation of France (Mme N. Blaize) and the Delegation of the USA (Mr. W. Dubbert) to act as rapporteurs of the Session.

#### MATTERS OF INTEREST TO THE COMMITTEE (Agenda Item 4)

8. The Committee had before it working paper CX/MAS 88/2 containing matters of interest arising from the Seventeenth Session of the Commission and from various Codex

sessions. The Committee noted and agreed that most of the matters included in the paper would be discussed in detail under the appropriate agenda items as suggested in the paper.

- 9. On the question of methods of analysis for aflatoxins being developed by the Codex Committee on Food Additives and Contaminants, the Committee was informed that it would be in the interest of gaining time if the Committee could agree to reviewing paper CX/FAC 88/18-add 1. The Committee requested the Working Group on Analysis to consider whether the paper could be reviewed at such a short notice.
- 10. The Committee was informed that the Commission had accepted that methods of analysis and sampling developed by the Codex Committee on Residues of Veterinary Drugs in Foods need not be endorsed by the Committee. It also noted that methods of analysis for pesticide residues, microbiological methods, and methods of analysis included in Codex specifications for food additives did not require endorsement by the Committee. The delegations of the United Kingdom and the Netherlands queried why such methods should not require endorsement. While it was noted that more frequent sessions of the Committee or its Working Group on Endorsement might result if more methods of analysis being referred to the CCMAS for endorsement, it was also noted that it would not be possible to arrange for such a working group to meet because these meetings would have to follow all Codex rules on meetings. It was pointed out that analytical methods for pesticide residues, veterinary drug residues and microbiological methods represented specialized fields which might explain why such methods were not being referred to the Committee for endorsement.
- 11. As regards the question of updating references to Codex methods of analysis and sampling and also of including references to identical methods published by other organizations than those to which the Codex methods referred, the suggestion was made that lists of Codex methods indicating all appropriate references could be prepared and published for the information of laboratories. The burden of updating references and communicating them to the Codex Secretariat rests with the Organization concerned. The use of the Swedish Food Laboratory Newsletter and the AOAC Bulletin "Referee" were also envisaged for this purpose.
- 12. The delegation of the Netherlands stated that a collaborative study on the determination of copper, iron and nickel in fats and oils had been completed by IUPAC. The report of the collaborative study was made available to the Committee.

#### NATURE AND PURPOSE OF CODEX METHODS OF SAMPLING (Agenda Item 5)

- 13. The Committee had before it documents CX/MAS 88/3 prepared by the Secretariat on the nature and purpose of Codex methods of sampling. The paper analysed the task of Codex committees in the elaboration of sampling plans and other recommendations related to sampling on the basis of the General Principles for the Establishment or Selection of Codex Sampling Procedures (Procedural Manual of the Codex Alimentarius Commission, 6th edition). The Secretariat had concluded that the General Principles required the development of detailed sampling procedures for the various Codex standards and that this task appeared to be potentially very extensive for the various Codex standards. The question of whether Codex sampling procedures should be advisory or mandatory (i.e. subject to acceptance by governments) was not entirely clear. The Secretariat was of the opinion that the CCMAS should discuss this question and also which aspects would require internationally agreed upon procedures in relation to consignments of food moving in international trade.
- 14. The Chairman of the Working Group on Sampling, Dr. R. Wood (UK), reported on the conclusions of the informal discussions on sampling (see para. 6). The Working Group had concluded that it would be preferable to develop recommendations on sampling through a single Codex document, rather than approaching sampling on a Codex standard by standard, i.e. commodity by commodity, basis. Such a Codex document should include technical administrative and other appropriate recommendations aimed at assisting in the interpretation of compliance with Codex standards. A reissue of a questionnaire requesting information on the approach to sampling in individual countries also might be desirable.

- 15. The delegation of the Netherlands queried why sampling procedures to be elaborated by the Codex Committee on Residues of Veterinary Drugs in Foods should not be subject to endorsement by the Committee. It was noted by the delegation of the USA that such a procedure would require the inclusion of appropriate experts in the delegations attending sessions of the CCMAS.
- 16. The Committee discussed the approach to sampling adopted by the Commission for pesticide residues in food. It noted that the approach was a practical one and defined compliance on the basis of the analytical sample. The opinion was expressed that such a legal, albeit practical approach had its disadvantages in that it disregarded the heterogenicity of the lot. It was also noted that the sampling approach for pesticide residues did not include a provision for defending the interests of the exporter in cases of dispute. The delegation of Switzerland indicated that a more elaborate plan would be used in its country in case of disagreement concerning the results of analysis. The delegation of the USA was of the opinion that the Codex sampling procedure for pesticide residues represented a too simple approach in which, by definition, the sample represented the lot and that any problems concerning the results related only to analytical errors. There was a need to reconcile such practical approaches to sampling with statistically sophisticated sampling plans. The opinion was expressed by several delegations that Codex should develop sampling plans at two levels of inspection.
- 17. The Committee <u>agreed</u> that a single Codex document on sampling should be developed rather than including sampling provisions in individual Codex standards. It invited the Working Group to continue discussing this question further with a view to developing recommendations as to the contents and status of such a Codex document, "A Guideline on Sampling and Related Matters" (see also para. 23).

### GUIDELINES ON SAMPLING - REVIEW OF CODEX STANDARDS WITH REGARD TO SAMPLING (Agenda Items 6 and 7)

- 18. The Committee had before it documents CX/MAS 88/4 and 88/5 prepared by the Inter-Session Working Group on Sampling. The document of that session had been reworked by the Secretariat and presented as two separate documents. The Chairman of the Working Group, Dr. R. Wood, expressed the opinion that it would have been preferable to present the conclusions of the Working Group in a single document as prepared by the Working Group itself. Dr. Wood also expressed his disappointment that Codex Committees had taken little action on the Sampling Instructions.
- 19. The delegation of Switzerland was of the opinion that duplication of effort should be avoided in the preparation of Codex Guidelines on Sampling. He drew particular attention to the FAO Manual of Food Quality Control which was in preparation. It was noted that the FAO Manual No. 9 Introduction to Food Sampling would be issued shortly. This manual would deal mainly with sample procurement and other aspects including the size of the sample to be taken for a number of food products. It would not address the question of lot acceptance criteria. Any material in the FAO Manual which would be useful for discussion by the Committee could eventually be included in the proposed Codex Guidelines on Sampling.
- 20. On the question of what information might be included in the proposed Codex Guidelines on Sampling, the Committee agreed that the Guidelines should not contain material which is readily available in published texts, including the FAO Manual. Rather, the Codex Guidelines should address questions which required internationally agreed recommendations. The Guidelines would also include the technical recommendations concerning sampling included in the present Sampling Instructions for Codex Committees (CX/MAS 1-1987). In particular, the proposed Guidelines should address lot acceptance criteria for the various provisions included in Codex Standards requiring analysis. The Codex Guidelines on Sampling would also be the appropriate publication in which specific sampling instructions developed by Codex Committees could be included.
- 21. The Committee considered in detail the recommendations of the Working Group on Sampling which had met during the session (see para 20) regarding the contents of the proposed Codex Guidelines on Sampling. The list of contents agreed upon by the Committee for the proposed Codex Guidelines is given in Appendix II. It was confirmed

that it would be preferable to approach sampling through a single advisory document to which reference could be made in individual Codex Standards. The Committee was led to this conclusion because of (a) lack of consistency in approach to sampling by Codex Committees, (b) lack of response to the request that Codex Committees consider the question of sampling on the basis of the Codex Instructions on Sampling and (c) because most provisions in Codex Standards, in particular compositional characteristics, would require a common approach to sampling and lot acceptance.

- 22. The Committee recognized that in certain cases, especially with attributes such as visual defects and some special health related problems (e.g. aflatoxins in food, microbiological end product specifications, etc.), Codex Committees dealing with the subject matter would have greater expertise and should be responsible for developing sampling procedures including lot acceptance criteria for inclusion in the Codex Sampling Guidelines.
- 23. The Committee requested the Commission to give consideration to the approach proposed above which would involve the development of a single advisory Codex document on sampling with inputs from Codex Commodity and other Codex Committees. Should the Commission decide to proceed as proposed by the Committee, it was agreed that it would be necessary to arrange for a first draft of the document to be prepared and circulated for comments. The Committee expressed its willingness to consider such a Codex document on sampling on the basis of comments (see Appendix II). The delegations of Hungary, USA, UK and others expressed their interest in participating in work on the sampling document.

#### **SAMPLING FOR NET CONTENTS** (Agenda Item 8)

- 24. The Committee had before it a short working paper (CX/MAS 38/6) informing the Committee of the status of the OIML Draft International Recommendation on Sampling for Net Content in Packages. It also had before it the OIML sampling plans as adopted by the 8th International Conference of Legal Metrology (Sydney, October 1988). The delegate from the USA (Dr. A. Rainosek) provided the Committee with a description of the statistical characteristics of the variables sampling plans included in the OIML document. The Committee agreed that it would discuss only the variables plans, not the attributes plans, since the Codex General Standard on Labelling specified that enforcement of net contents be done on the basis of "The average content of the units of the lot shall be equal to or greater than the declared contents."
- 25. The Committee noted that the OIML recommendation included two sampling protocols to determine compliance with declared net contents a) attribute plans for the percentage of non-conforming units in a lot and b) variables sampling plans for the mean value of a lot. Dr. Rainosek explained that the variables sampling plans contained statistical provisions which offered adequate protection to the producer and consumer against making incorrect decisions concerning lot disposition. These plans, apparently, had been selected assuming non-destructive sampling. Lot sizes smaller than 150 packages were not addressed. Being general statistical plans, they were applicable to prepackaged foods, whatever the content of the container. The sample size given for lots containing more than 4000 packages offered an even higher degree of protection to the consumer and this was considered to be in conformity with international convention. However, the OIML recommendation made provision for breaking down large lots into smaller lots, should this be deemed desirable.
- 26. The question was raised as to how lots containing less than 150 packages should be inspected. It was noted that one approach would be to resort to 100% sampling or to amend the OIML sampling recommendation to specify a lower limit of lot size. The delegate from France drew attention to section 2.3 "Storage Effects", and queried how any changes caused by storage would be recognized in the evaluation of net contents. In the discussion it was also queried as to how the sampling plans would recognize overfill which may be significant in the case of certain types of product. The Committee noted that the OIML sampling plans were one-sided and covered only short weight. The question was raised why an attribute type of sampling plan could not be used. The Committee noted that this was certainly possible, but that the General Labelling Standard specified the "average" thereby requiring a variables plan (see also para. 24).

- 27. The Committee noted that the Codex General Labelling Standard did not specify an average or any other type of sampling strategy for drained weight and that the determination of this characteristic would involve destructive sampling. The Committee agreed that a sampling plan for drained weight should be developed and that the OIML recommended variables plans may not be applicable in view of the relatively high rates of sampling. Concerning the question of destructive sampling, the delegation of France indicated that, with a number of types of containers such as bottles, destructive sampling was required since such containers could not be assumed to be of uniform weight.
- 28. The Committee also noted that sampling for net weight varied from country to country and that there were several valid statistical approaches to check compliance with net contents provisions. It was, therefore, agreed that it would be necessary to obtain comments from governments and from International Organizations on the OIML sampling plans before finalization and endorsement as a Codex recommendation.
- 29. The Committee decided that the OIML sampling plans represented a good basis for discussions on the control of net contents in lots moving in international trade. The OIML sampling plans should, therefore, be submitted to the Commission with the request that it should be referred to governments and International Organizations for comments in accordance with an appropriate procedure (see Appendix V). The views of the Codex Committee on Food Labelling and appropriate Codex Commodity Committees should also be sought. The Secretariat should obtain answers to specific questions from the appropriate bodies, e.g. the question of drained weight, the one-sided feature of the OILM sampling plans etc. The Committee also agreed that, should the Commission decide to proceed with the elaboration of sampling plans for net contents, the CCMAS would be the appropriate Committee to consider these sampling plans. The Committee requested the US delegation (Dr. Rainosek) to act as rapporteur on this topic and to study any comments and information received and to report to the next session of the Committee.

#### ENDORSEMENT OF METHODS OF SAMPLING IN CODEX STANDARDS (Agenda Item 9)

30. The Committee had before it working document, CX/MAS 88/7, containing a proposed revised sampling plan for food grade salt and working papers summarizing the sampling plans requiring endorsement by the Committee, CX/MAS 88/8 and CX/MAS 88/8 Add 1.

#### Sampling plan for food grade salt

- 31. The working paper on food grade salt was introduced by Dr. J.M. Rafols of the ECSS. He informed the Committee that the revised sampling plans still required editorial improvement, especially in the French version. Furthermore, the question of the number of laboratory samples to be analysed (see Section 7.1) still required discussion since it was thought that a lesser number of analyses could be carried out.
- 32. Following detailed discussions, the Committee <u>agreed</u> that the sampling plans should be revised by a drafting group consisting of <u>persons</u> familiar with sampling procedures for salt. The delegations of USA, Poland, France and the Representative of the ECSS were requested to examine the sampling plans.
- 33. On the recommendation of the drafting group, the Committee <u>adopted</u> the changes given below to the sampling plan for salt. It was also agreed to <u>make editorial changes</u> in paras 3 to 5 to make these consistent with the changes adopted below.
  - (a) It was agreed that para 3 (d) of the Introduction of CX/MAS 88/7 should be amended by deleting the last four lines of the text beginning with "Because the large...".
  - (b) Section 6 Procedure in Section 6.1.2 the word "periodic" was deleted in the title as well as in the text of this paragraph.
  - (c) Section 6.3.2 was changed to: "One or more blended bulk samples, each composed of a portion of the items drawn from the lot, form the laboratory

- sample". It was noted that "laboratory sample" would correspond to the "blended bulk sample" described and represented in CX/MAS 1-1987 Appendix IV page 19, para 4.
- (d) Section 6.3.3 was deleted and replaced by "At least 2 test portions of the laboratory sample are analysed". The Committee believed that using n=2 or n=3 rather than n=5 as presently given in the document, would not adversely affect the intent of the method or its precision. It was also noted that it was current practice to use only 2 duplicates for replicate laboratory analyses.
- (e) Regarding the Annex, which included examples on manimum sample size, the Committee decided to delete it and to include reference to the Codex Instructions on Sampling (CX/MAS 1-1987 Appendix V, Table 3), as follows: "The number of items to be inspected should be the number specified for the size of the specific lot for inspection level 2 in Table 3, CX/MAS 1-1987 Appendix V."
- 34. The Committee endorsed the sampling plan for food grade salt, as amended (see Appendix III), for the determination of essential composition and quality factors in Section 3 of the standard. It was agreed that, at a later stage, the sampling plan might be incorporated in a general Codex publication on sampling. It was noted that the ECSS might also wish to publish the sampling plan following its adoption by the Codex Alimentarius Commission.

#### Sampling Plans included in Draft Codex Standards

35. The delegation of the United Kingdom expressed its concern that little action had been taken by Codex Commodity Committees to review sampling plans included or to be included in Codex standards on the basis of the Instructions on Sampling. The Committee agreed that it was necessary to consider sampling in Codex standards and draft standards on the basis of the Instructions and requested the Secretariat to take appropriate action.

#### Unshelled pistachio nuts and dates

36. The Committee noted that the sampling procedure recommended by the Commodity Committee did not include any provisions for lot acceptance criteria or any detailed recommendations concerning the size of the sample in relation to lots. The remark was made that information was required on aflatoxin producing moulds in pistachio nuts in relation to the acceptability of the lot. The Committee did not endorse the proposed sampling procedures and invited the Commodity Committee to reconsider the matter in the light of the Sampling Instructions.

#### Vegetable protein products, soy protein products, wheat gluten

37. The Committee noted that the Commodity Committee had not taken into account the Sampling Instructions and that the ISO methods (2170-1980, Sampling of Milled Products) included detailed instructions on sample taking and sample size, but did not include lot acceptance criteria. It was decided to endorse the ISO sampling procedure on a temporary basis pending review of the question of sampling both by the Commodity Committee and the CCMAS.

#### Standards and draft standards being elaborated by the Coordinating Committee for Africa

38. Noting the remarks made in connection with vegetable protein products (see para 37), the Committee postponed endorsement of the ISO and ICC sampling procedures included in these standards. The Secretariat was requested to bring the conclusions of the Committee regarding sampling to the attention of the Coordinating Committee for Africa.

#### Quick frozen fish fillets and squid, dried shark fins

39. The Committee noted that these standards included reference to the Codex attribute sampling plan (CAC/RM 42-1969) for the verification of visual defects and net contents. It also noted that, in the case of quick frozen squid, the Codex sampling plan developed for quick frozen fish blocks had been recommended for blocks intended for further processing. Considering that the standards concerned were at early Steps in the Codex procedure and that the question of sampling would be reviewed both by the Commodity Committee and the CCMAS, it was decided to endorse the sampling plans only on a temporary basis.

Draft standards for pulses, sorghum grains and flour, durum wheat flour and semolina, wheat flour, maize, whole maize meal, degermed maize meal and maize grits

40. The Committee noted that the Commodity Committee had recommended that the sampling procedures included in these draft standards be temporarily endorsed. The Committee agreed to this proposal. It noted that the various references included in the above draft standards represented mainly sampling procurement methods, with some recommendations concerning sample size, but did not conform with the recommendations included in the Sampling Instructions. For example, no lot acceptance criteria had been recommended. The Commodity Committee was requested to give this matter further consideration.

#### Sampling for aflatoxins in cereals, pulses and legumes

41. The Committee noted that the Commodity Committee had considered the question of sampling for aflatoxins on the request of the Codex committee on Food Additives and Contaminants (CCFAC) and had recommended a simple practical approach based on the average of the lot determined on a single randomly selected composite sample. It was also noted, however, that such an approach would require a change in the value of the guideline level proposed for aflatoxins by the CCFAC. The Committee welcomed such a joint effort in arriving at practical solutions to sampling but also noted that further information was required on the distribution of aflatoxins in cereals, pulses and legumes so that a statistical sampling plan could be developed. It was agreed that the interim practical approach suggested by the Commodity Committee could be temporarily endorsed but that additional comments were needed.

#### Soy bean oil

42. The Committee expressed its satisfaction at the efforts of the Commodity Committee in applying the Sampling Instructions. However, it was noted that a specific sampling plan was being developed for net contents (see paras 24 to 29). It also noted that further consideration would have to be given to the question of lot acceptance for food additives and to the development of Codex sampling guidelines (see paras 18 to 23). The Committee, therefore, decided to temporarily endorse the proposed sampling procedure for soy bean oil and invited the Commodity Committee to keep this matter under review.

#### Environmental contaminants

43. The Committee noted that the CCFAC had suggested that the sampling procedure developed for pesticide residues (CAC/PR 5-1988) might also be applicable for lead, cadmium and mercury. It also noted that this approach corresponded to a sampling procedure involving the analysis of a blended bulk sample (see Sampling Instructions, Appendix IV, B). The delegation of the UK expressed the opinion that the heterogenicity of food with respect to this environmental contaminants might not permit the use of such a simple sampling plan. The delegation of the USA informed the Committee that the distribution of residues resulting from the application of pesticides was relatively well-known, but that information on the distribution of environmental contaminants in food was lacking. More information was required before an opinion could be formulated on the applicability of such a sampling approach as proposed by the CCFAC. The delegation of Hungary did not share this view and expressed the opinion that both toxicological considerations and considerations of homogeneity would permit a composite sample approach to be followed in case of the environmental contaminants mentioned. The

delegation of the Netherlands indicated that the distribution of environmental contaminants in food (skewed distribution) would not permit a simple sampling approach. The delegation of Denmark informed the Committee that the level of mercury in fish depended on the size of the fish and that this contributed to the problem of setting limits and recommending sampling procedures. The delegation of Australia indicated that the uneven distribution of contaminants, even within a product, might require further consideration.

44. The Committee considered that it would not be possible to give a definite opinion on the applicability to environmental contaminants of a simple sampling procedure such as that proposed for pesticide residues, without a knowledge of the distribution in food of the contaminants in question. The CCFAC was invited to give this question further consideration, including the part of the animal and plant products to which the quideline levels applied.

#### REPORT OF THE SEVENIH INTER-AGENCY MEETING (IAM) (Agenda Item 10)

- 45. The Committee had before it the report of the Seventh Inter-Agency Meeting (Conference Room Document 1). The report was introduced by the secretary of the IAM, Mr. K.G. Lingner (ISO). In his report Mr. Lingner highlighted the decisions reached by the IAM and noted that good progress had been made by international organizations in responding to the requests of the Codex concerning the development or testing of methods of analysis. As regards sampling, the IAM had noted that further advice would be forthcoming from the CCMAS in this field and had decided, therefore, not to take further action. Significant progress had been made in the harmonization of the protocol for collaborative testing of methods. He expressed the opinion that it would be essential to include the report of IAM in the report of the CCMAS and requested the Codex Secretariat to explore the possibility to make this possible.
- 46. The Committee was in unanimous agreement with the request of the Secretary of the IAM, supported by its Chairman, Mr. G. Castan (ISO), that it would be highly desirable to include the IAM report as an appendix of the report of the CCMAS, as in the past.
- 47. A member of the ISO delegation to the IAM (Dr. de Ruig, the Netherlands) informed the Committee about a new approach for the validation of methods (by the application of criteria for proving the presence of an analyte in a matrix, instead of through meticulously described methods). He informed the Committee that, in order to prove the presence of an analyte, such criteria had been specified for a number of analytical methods and that these had been included in a draft EEC document (VI/1541/88EN) entitled "Criteria for Reference Methods of Analysis for Residues".
- 48. Regarding paragraph 10 of the IAM report, the representative of the AOAC informed the Committee that a further meeting of the IUPAC Working Group on Harmonization would be held in Washington, DC, USA from 17 to 20 April 1989 on the harmonization of quality schemes in chemical analysis and on the adoption and presentation of analytical methods standardizaed by collaborative studies (see para 52).
- 49. The Committee agreed that it would be necessary to identify priorities and to identify the needs of Codex for methods of analysis and that this was a task for the CCMAS with the assistance of the Secretariat (see para 27 of the IAM report).
- 50. Regarding the question of the role of International Organizations in developing sampling methods for food products, the Committee <u>agreed</u> that it would be in a better position to identify such a role following discussions on Codex sampling procedures (see para 29 of the IAM report).
- 51. The Secretariat informed the Committee that the general method for ashing proposed by the delegation of the USSR at the last session of the Committee had been referred to the IAM for further consideration. This had been done because, in the opinion of the Secretariat, the development of a general ashing procedure would be a task more appropriate to an international organization than to the CCMAS. The delegation of the USA indicated that, following consultation with the delegate from the USSR, a re-edited version of the general method had been prepared for publication in the JAQAC. Comments

would be received on the general method of ashing which could be considered by a working group on analysis of the CCMAS at its next session. A suitable note of information should be included in the AOAC Bulletin "Referee" as well as in the Swedish Food Laboratory Newsletter. The Committee was in agreement with this procedure. The delegate from the USSR was also in agreement, especially since in his opinion, it would be difficult to design a collaborative test for such a general ashing procedure (see paras 32 to 35 of the IAM report).

- 52. The Committee noted the view of the Codex Secretariat expressed at the IAM that there was a need for collaboration between the various International Organizations in the development of a defining method for a given characteristic in a particular food product (see para 60 of the IAM report). It also noted that, given the large number of methods required for the analysis of food additives in foods, priorities would have to be set and that there would have to be close collaboration among International Organizations in the testing of such methods (see para 65 of the IAM report).
- 53. The Committee expressed its appreciation to the IAM as well as to its Chairman, Mr. Castan, and its secretary, Mr. Lingner, for the valuable contribution to the work of the Codex Alimentarius Commission in the field of sampling and analysis. It noted that the IAM would continue to meet prior to sessions of the CCMAS. The report of IAM is given in Appendix VIII to this report.

#### CLASSIFICATION OF CODEX METHODS OF ANALYSIS (Agenda Item 11)

- 54. The Committee had before it a working paper prepared by the delegation of the UK (CX/MAS 88/9) on problems relating to the classification of Codex methods into "defining", "reference" and "alternative approved" methods. The paper gave examples where the classification of a Codex method had been inconsistent or where it had been difficult to interpret the General Principles for the Establishment of Codex Methods of Analysis. For example, it was not clear whether the determination of protein through the use of an agreed factor, and the determination of nitrogen should be considered as Type I or Type II. The correct classification of Codex methods was considered to be essential in view of the obligation falling on governments in accepting Codex standards containing such methods.
- 55. The delegation of the USSR suggested that, in principle, Codex methods should be classified on the basis of metrological data relating to the performance of the method. The opinion was expressed that too much emphasis was being given to the classification of Codex methods. On the other hand, it was thought that for the purpose of classification, clear guidance was necessary especially for Codex Committees. Regarding the need for collaborative studies for "old" methods which had been classified as Type IV, the representative of IFG indicated that it would be possible to perform some of the additional tests required. The Committee noted that the use of commercial test kits might cause difficulties if such methods were assigned a Codex status, since the composition of such kits could vary, or the availability of the kits might decrease because of commercial reasons.
- 56. The delegation of the USA expressed the opinion that the classification of methods was working generally well, but that some guidelines might be useful so that the classification could be done in a consistent manner. The USA delegation also expressed concern that countries did not have time to study in detail the paper prepared by the United Kingdom and that it should be distributed for comments. The delegation of Canada expressed the opinion that the difference between Type I and II methods was more apparent than real since both methods were designated Codex methods which were intended to be used in cases of dispute.
- 57. The Committee <u>decided</u> that the document prepared by the United Kingdom (see Appendix VI) should be sent to governments for comments, and that the question of classification including the concept of simple methods, should be rediscussed at the next session. The delegation of the UK agreed to study the comments and to prepare a concise set of guidelines for the classification of Codex methods of analysis.

#### LIMIT OF DETERMINATION (Agenda Item 12)

- 58. The Committee had before it a document CX/MAS 88/10 prepared by Hungary on the question of the definition of "limit of determination". The paper, which was introduced by the Hungarian Technical Secretary (Dr. A. Zsigmond), outlined the discussions which had taken place on this subject at previous sessions of the Committee, including the proposal of the delegation of the USSR, at the Fifteenth Session of the Committee, that a new and more appropriate definition should be given to quantify the concept of limit of determination.
- 59. In response to a query from the delegation of the Netherlands, the Secretariat explained that the scientific material submitted by the delegations of the USSR and Czechoslovakia at the last session had not been distributed for governments comments as requested by the Committee. The reason for this decision by the Secretariat was that it was considered that the material in question would be more appropriately discussed by interested International Organizations such as IUPAC, AOAC or ISO. For this reason the IAM had been requested to consider the matter (see para 81, Report of the Seventh Inter-Agency Meeting). The Secretariat also stated that the CCMAS would probably be more interested in the results of collaborative tests in which the limit of performance of the method was indicated rather than discussing how the limits of determination or detection might be defined in precise mathematical terms. The Netherlands drew the attention of the Committee to a paper on Recommendations for the Definition, Estimation and Use of the Detection Limit, Royal Society of Chemistry, Burlington House, Piccadilly, London WIV OBN, UK. Analyst, February 1987 Vol. 112.
- 60. The delegation of Czechoslovakia suggested that ISO TC 69 should discuss the question raised above. The representative of ISO stated that the terms of reference of TC 69 SC 6 would be too broad to be of assistance in this matter.
- 61. The delegation of the USA informed the Committee that the new proposed definition of the limit of determination relating to considerations of safety would be submitted to the JACAC for publication in the form of a scientific paper prepared by Professor I. Skurihin (USSR).
- 62. The Committee <u>agreed</u> that it would be more appropriate to refer the matter to IUPAC for evaluation and requested the Secretariat to collect all papers and scientific material which had been put before the Committee in the past and to transmit them to the IUPAC with the request that the question be considered by that Organization. The Committee wished to be kept informed of developments.

#### SELECTION OF SIMPLE METHODS OF ANALYSIS (Agenda Item 13)

- 63. The Committee had before it a working paper (CX/MAS 88/11) prepared by the Codex Secretariat at the request of the Committee and the Commission. The paper explored ways and means of assisting developing countries in the selection of "simple" methods of analysis.
- 64. The Committee noted that the Codex Committee on Pesticide Residues had defined "simple" methods with reference to certain types of instruments, reagents etc. The delegations of the Netherlands and Cuba were of the opinion that HPLC determinations did not reflect a simple method. The delegation of the UK was not convinced that it was the task of Codex to propose simple methods of analysis, and also expressed the view that the suggestion of the Secretariat concerning a list of equipment, materials, etc. suitable for use in laboratories in developing countries be prepared; would not be an appropriate task for this Committee. The UK delegation further stated that such information may already be available in other FAO documents. The delegation of Australia informed the Committee that a WHO group concerned with narcotic drugs had prepared a list of chemicals, equipment and laboratory designs along the lines suggested above and that this material may be relevant to the analysis of food.
- 65. The delegation of Cuba expressed its satisfaction with the document which addressed a problem facing many developing countries, and that the issue had been discussed for many years. The delegation suggested that the document be circulated to

governments and that action should be taken to assist laboratories with limited facilities.

66. The Committee agreed that the definition of "simple" methods of analysis developed by the CCPR might not be applicable to methods of analysis generally; especially with respect to HPLC determinations. It was decided to refer the working paper prepared by the Secretariat to Codex Committees including Codex Coordinating Committees for appropriate action. It also agreed with the suggestion that a list of equipment, materials, etc. along with minimum purity requirements for reagents for laboratories would be useful to developing countries, but that this work would be more appropriate for the FAO rather than the CCMAS.

#### ENDORSEMENT OF METHODS OF ANALYSIS IN DRAFT STANDARDS (Agenda Item 14)

- 67. The Committee had before it working papers containing methods of analysis subject to endorsement by the Committee, supporting information submitted by the ECSS and on the determination of total fat content and egg-yolk content in mayonnaise (CX/MAS 88/12 Parts II and III, CX/MAS 88/12 Add 1) and the report of the ad hoc working group on analysis (Conference Room Document 2). The report of the working group was introduced by its Chairman (Dr. W. Horwitz, USA). Dr. Horwitz indicated that the working group had studied the proposed methods in detail, including those methods which had been reviewed by various Codex Commodity Committees. He indicated those points where the working group had requested further information before a decision could be reached concerning endorsement.
- 68. The Committee discussed the working papers and the report of the working group. The following remarks were made during the discussions:
  - (a) With reference to the determination of lead in cocoa products and edible fats and oils, the representative of IUPAC requested that a reference be included in the report indicating that a collaborative study of a method for lead in fats and oils had been completed and would be published in the near future.
  - (b) Regarding the method for loss on drying in certain standards for foods for special dietary uses the representative of IFG suggested that the Commodity Committee be requested to consider drying of high fructose products at  $70\,^{\circ}\text{C}$  in vacuo.
  - (c) A number of delegations were of the opinion that the temporary endorsement proposed by the working group for the determination of vitamins D and E for specified vegetable fat products be changed to full endorsement. The Commodity Committee should be requested to propose more modern methods for consideration by the Commodity at a future session.
  - (d) The delegation of the USSR recommended that the Committee should request the International Organizations to send, prior to the next session of CCMAS, information on the results of metrological investigations for all methods to be endorsed.
- 69. The Committee <u>adopted</u> the report of the Working Group on Analysis with the above amendments (see <u>Appendix IV</u>). The Committee expressed its appreciation to Dr. Horwitz, Chairman of the Working Group and the Secretariat for their valuable contribution in evaluating the methods proposed by Commodity Committees.

#### GENERAL METHODS OF ANALYSIS FOR CONTAMINANTS (Agenda Item 15)

70. The Committee noted that document CX/MAS 88/13 was not available but that information had been received from the IDF on the determination of copper content in milk and milk products (international IDF standard 76A:1980) using a photometric procedure with diethyl dithiocarbamate. Information had also been received during the session (see para 12) from the IUPAC representative on the determination of copper, iron and nickel in fats and oils using a graphite furnace AAS method. It was noted that these methods were not general methods. The delegation of Hungary also informed the

Committee that the method for the determination of copper on which comments had been received (AOAC XIV, 1984, 25.066, carbamate method) had been endorsed for use in countries of the CMEA.

71. The Committee <u>agreed</u> with the suggestion that it was timely to review the Codex general methods for contaminants and decided that the general methods so far adopted by the Commission, together with the proposed alternative approved method for copper proposed by the USSR, be circulated for comments and that the methods be reviewed at the next session (see Appendix VII).

#### METHODS OF ANALYSIS REQUIRED BY CODEX (Agenda Item 16)

- 72. The Committee was informed that working paper CX/MAS 88/14 containing a list of methods of analysis requiring either to be developed or validated had been considered in detail by the IAM (see para 8). The Secretariat explained that the list of methods included in this document was not exhaustive since it did not include methods required for the determination of contaminants, food additives and other such analytes of general application to food. The Secretariat also expressed the view that it was up to the CCMAS and interested Codex Committees to state their exact request for methods which should be developed or tested by International Organizations. There was a need for stating priorities, especially for methods of analysis for food additives in food which represented a considerable work load.
- 73. Noting that the methods of analysis listed in document CX/MAS 88/14 had been discussed by the Working Group on Analysis, the Committees did not discuss the individual methods any further. However, it agreed with the Secretariat that Commodity Committees should be requested to indicate their needs for methodology taking into account the importance of the provision or of the food commodity so that the CCMAS would be in a position to request action by International Organizations on methods of importance for the Codex Alimentarius Commission. The Chairman of the Committee expressed the opinion that the determination of micro-elements and vitamins would represent a priority.

#### FUTURE WORK (Agenda Item 17)

- 74. The Committee noted that it had sufficient future work in the endorsement and review of methods of analysis and sampling and in the consideration of special tasks relating to sampling and analysis, as indicated below.
- 75. The Committee was informed that a special volume of the Codex Alimentarius might be devoted to questions relating to analysis and that the Secretariat hoped to be in a position to report on developments to the next session of the Committee. This volume of the Codex Alimentarius would consolidate appropriate material available on analysis in the various Codex publications and reports, including the general methods so far adopted.
- 76. The Committee noted that the following represented ongoing and future work:

#### Sampling

- Endorsement of sampling provisions in Codex standards
- Endorsement and review of specific sampling plans elaborated by Codex Committees
- Sampling plans for net contents
- Sampling plans for drained weight
- Codex guidelines on sampling and related matters
- Consideration of questions related to sampling arising from the IAM

#### Analysis

- Endorsement/review of methods of analysis in Codex standards
- Elaboration/review of Codex general methods of analysis for contaminants and other general methods, e.g. food additives
- General method of ashing for the determination of heavy metal contaminants

- Specific analytical questions referred to the Committee for consideration
- Consideration of "simple" methods and selection of further alternative approved methods (including simple methods)
- Guidelines for the classification of Codex methods
- Matters arising from the IAM

#### OTHER BUSINESS (Agenda Item 18)

- 77. The delegation of Cuba expressed the wish that all efforts be made to provide translation of all working papers into Spanish and to provide interpretation into that language. In the opinion of Cuba, this would contribute greatly to a more active and fruitful participation in the work of the Committee by Spanish speaking countries.
- 78. Following consideration of a written statement by the delegation of Cuba and statements by various delegations as well as by the Secretariat, the Committee expressed its desire that working papers be distributed well in advance of sessions of the CCMAS so that governments have ample opportunity to study the technical papers.
- 79. Dr. W.G. de Ruig of the Netherlands introduced a room document (CX/MAS 88/19) on the "use of criteria in analytical control" and a draft EEC document (VI/1541/88-EN) on "Criteria for Reference Methods of Analysis for Residues". These two papers presented a new approach for the validation of analytical methods. The Committee had a brief discussion of the approach proposed in these papers and noted that Dr. de Ruig's paper would be published in the Journal of Chemometrics. The Secretariat informed the Committee that the recent session of the Codex Coordinating Committee for Europe had also discussed chemometric methods for the identification of food products and the determination of food ingredients. The delegation of the USA expressed the opinion that while these methods were interesting they did not address the problem of variability.
- 80. The Committee expressed its interest in the new methods mentioned in para 79 above and hoped that this new scientific development would be given adequate attention by the scientific community. It wished to be informed of future developments.
- 81. The delegation of Czechoslovakia expressed the opinion that methods should all use the international adopted IS units of measurement and terminology.

#### TIME AND PLACE OF NEXT SESSION (Agenda Item 19)

82. The Committee was informed that the Seventeenth Session of the CCMAS would be held in Budapest probably during the end of 1990.

#### SUMMARY STATUS OF WORK

Subject	Action to be taken by:	Document Ref. (ALINORM 89/23)
Guidelines on Sampling	CAC Hungary, USA, UK Governments	para. 23, App. II
Sampling for Net Contents	CCMAS  CAC Governments USA	para. 29, App. V
Sampling Plan for Food Grade Salt	CCMAS CAC	para. 34, App.
Sampling Plans for various Codex commodity standards	CCPFV CCVP CC/AFRICA CCFFP CCCPL CCFO CCFAC	para. 36 para. 37 para. 38 para. 39 paras. 40-41 para. 42 paras. 43-44
Guidelines for classification of Codex methods of analysis and "simple" methods of analysis	UK Governments CCMAS	paras. 54-57, Appendix VI
Definition and selection of "simple" methods of analysis	Codex Committees	paras. 63-66
Review of Codex General Methods of Analysis for Contaminants	CAC Governments CCMAS	paras. 70-71, Appendix VII
Methods of analysis needed by Codex - definition of priorities	Secretariat Codex Committees CCMAS IAM	paras. 72-73

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#### OUTLINE OF DRAFT CODEX GUIDELINES ON SAMPLING AND RELATED MATTERS

#### 1. Introduction

- 1.1 Purpose and scope
  - Codex Manual of Sampling
  - Harmonization across Committees
  - Collection of Codex approaches to sampling
- 1.2 Description of contents
- 1.3 History of Codex Sampling
- 1.4 Codex Acceptance Procedures including relationship between acceptance of methods of sampling and sampling procedures
- 1.5 General principles on sampling and explanatory notes
- 2. <u>Instructions on sampling procedures</u>

This section would include an appropriately amended text based on the Sampling Instructions CX/MAS 1-1987 including definitions of terms, etc.

- 3. Special considerations
- 3.1 The definition of "lot", "inspection lot" and "consignment" and discussion of special problems in identifying lots
- 3.2 Problems on handling a consignment consisting of mixed and intermingled lots
- 3.3 The heterogeneous nature of the distribution of many contaminants in a lot
- 3.4 Guidance for relaxing demand to obtain random sampling of items from a bulk shipment because of practical problems
- 3.5 Clarification of the point of sampling
- Re-sampling procedures to be followed following any dispute over the results of analysis
- 3.7 Considerations about homogenizing individual items to form a blended bulk sample
- 3.8 Organization and references for random sampling of product
- 3.9 Standard lay-out and format of sampling reports, including the way a sample is to be handled and prepared, the sealing and labelling of a sample, the documentation required to accompany a sample and the availability of a sample for continuous inspection
- 3.10 Consideration of previous results obtained from one establishment when resampling is indicated
- 4. Lot acceptance criteria
  (specification of the sampling plan to be used in relation to the various characteristics to be verified grouped in an appropriate manner)
- 5. Sampling procedures presently prescribed in Codex standards

#### Introduction

The sampling plan for salt developed by CCFA is a practical sampling procedure: it may be identified as type 4, (modified according to the example in para. 5 of Appendix IV of the Sampling Instructions CX/MAS 1-1987) i.e. a variables sampling procedure for mean quality - blended bulk samples analysed. Acceptance criteria are based only on a single result, the average associated with the range.

#### METHOD FOR SAMPLING OF FOOD GRADE SALT FOR COMPOSITIONAL CRITERIA

#### 1. SCOPE

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

The criteria to be used for acceptance or rejection of a lot or consignment on the basis of the sample are also provided.

#### 2. FIELD OF APPLICATION

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

#### 3. PRINCIPLE

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

A blended bulk sample is produced and, in order to ensure that it is representative of the lot or consignment, it is reduced to a number of laboratory samples, each composed of a proportion of the items drawn from the lot or consignment to be analysed.

Acceptance criteria is on the basis that the average of the samples extracted from the lot must comply with the provision in the Standard.

#### 4. DEFINITIONS

The terms used in this sampling method refer to those in the "Instructions on Codex Sampling Procedures" (CX/MAS 1-1987).

#### 5. HANDLING

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

#### 6. PROCEDURE

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

#### 6.1.1 Random sampling

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

6.1.2 Systematic sampling

If the N units in the lot have been arranged in a certain order and can be numbered 1 to N, then a 1-in-k systematic sampling of n items samples can be obtained as follows:

- (a) Determine the value of k = N/n. (If k is not a whole number, then round to the nearest whole number).
- (b) From the first k items in the lot select one item at random and then take every  $k^{\mbox{\scriptsize th}}$  item thereafter.

Bulk sampling
For salt in bulk, the bulk is notionally divided into items; a lot with a total mass of m Kg is considered to be composed of m/100 items. In this case, it is necessary to draw up a "stratified sampling" plan appropriate to the dimension of the bulk and the sampling points are selected from all the strata in proportion to the stratum sizes.

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

#### 6.3. Constitution of the sample

- 6.3.1 The size and the number of the items forming the sample depend on the type of salt and the lot size. The minimum increment to be taken should be constituted in accordance with one of the following specifications according to the circumstances:
  - 250 g of the product;
  - one package when salt is prepackaged in 0.5 or 1 Kg packages.

If more than one increment is taken they must be evenly distributed in the items forming the lot. Examples of minimum sample sizes can be found in the document CX/MAS 1-1987, appendix V, table 3, taking into account the appropriate inspection level; see paragraph 8.4 of this document.

- 6.3.2 One or more blended bulk sample, each composed of a portion of the items drawn from the lot, form the laboratory samples.
- 6.3.3 At least two test portions of the laboratory sample are analysed.

#### 7. ACCEPTANCE CRITERIA

7.1 Calculate the mean of the measured values for the characteristic (% NaCl) in the n test portions of the laboratory sample using:

$$\frac{\sum x}{n}$$
 =  $\frac{\sum x}{n}$  (n = 2, minimum)

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

 $\overline{\times}$  minimum level specified for the characteristic

#### 8. SAMPLING REPORT

The sampling report should contain the following information:

- a) the type and origin of the salt;
- b) alterations of state of the salt (e.g. presence of foreign matter);
- c) the date of sampling;
- d) the lot or consignment number;
- e) the method of packing;
- f) the total mass of the lot or consignment;

- g) the number of packages and the unit mass, and whether the mass is given net or gross;
- h) the number of items sampled;
- i) the number, nature and original position of the increments;
- j) the number, the composition and the mass of the bulk samples;k) if applicable, the method of reducing the bulk sample;
- 1) the number, the composition and the mass of the laboratory samples and the method by which they have been obtained and conserved;
- m) the names and signature of the people who carried out the sampling.

#### 9. BASIC REFERENCE

Document CX/MAS 1-1987.

#### 10. REMARK

"Laboratory sample" is the "blended bulk sample" described and represented in CX/MAS 1-1987, Appendix IV, page 19, step 4.

#### REPORT OF AN AD HOC WORKING GROUP ON ENDORSEMENT OF METHODS OF ANALYSIS

1. The following members constituted the  $\underline{ad\ hoc}$  Working Group on Endorsement of Methods of Analysis:

W. Horwitz G. Diachenko J.F. Lawrence I. Skurihin K. Trawicka I. Boros M. Harkay M. Vámos B. Borszéki A. Zsigmond C. Dahl J.M. Vallejo J. Daenen B. Whitehouse H. Wallin H. Woidich P. Venetz R. Gerber A. Vidnes G. Castan E. Nagy B. Mandrou B. Larsson B.E. Jensen C. Usher R. Kirk P. Hendrikse E. Elkins A. Gross I. Kamishlian W. Sanitz

E. Casadei

J.-K. Lee

I.-S Song

T. Serio J.M. Rafols

D. Procházka

Ying Hua Yang

USA
USA
Canada
USSR
Poland
Hungary
Hungary
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Finland Austria Switzerland Switzerland Norway

Norway ISO ISO OIV Sweden Denmark UK UK

Netherlands

USA USA USA

Federal Republic of Germany

FAO/WHO Secretariat Czechoslovakia Republic of Korea Republic of Korea China, Peopl's Rep. of

Cuba ECSS

- 2. The Working Group, under the chairmanship of Dr. W. Horwitz had the following tasks to perform:
  - (a) to consider endorsement of methods of analysis postponed, CX/MAS 88/12-Part I
  - (b) to consider endorsement of methods of analysis in Codex standards, CX/MAS 88/12-Part II
  - (c) to consider endorsement of methods of analysis in Draft Codex Standards not previously considered by the CCMAS, CX/MAS 88/12-Part III
  - (d) revision of Methods of Analysis provisions in Codex Standards for Sugars, CX/S 88/MAS and comment forwarded to Dr. R. Wood, UK

- (e) to consider endorsement of methods of analysis in Draft Codex Standards -Determination of Total Fat Content and Egg-yolk, CX/MAS 88/12-Add.1
- 3. The status of endorsement of methods of analysis is contained in Appendix I, Part I, Part II and Part III.

#### 4. Revision of Methods of Analysis for Sugars

Dr. Wood, delegate from the UK, briefed the Working Group on the report by the UK Secretariat of the Codex Committee on Sugars "Revision of Method of Analysis Provisions in Codex Standards for Sugars", document CX/S 88/MAS. He noted that this document had received extensive comments from its circulation to governments and interested organizations and that those comments would be forwarded to ICUMSA, ISO and IFG by the UK Secretariat. Following review of CX/S 88/MAS, the WG decided to recommend the temporary endorsement of the methods elaborated in this document, pending review by ICUMSA and ISO. The WG also pointed out the need for ISO and ICUMSA to conduct collaborative studies prior to final endorsement by CCMAS, for the many methods which had not been studied. The classification of the polarization method for Codex Standard for White Sugar (CODEX STAN 4-1981) was changed to a Type II and that of the loss on drying method for Codex Standard for Fructose (CODEX STAN 102-1981) was changed to Type I. The Cuban delegation wished the temperature correction extension procedure in the determination of polarization for white sugar to be referenced to the Report of the 19th Session of ICUMSA, 1986.

5. Endorsement of Methods of Analysis for Total Fat Content and Egg-Yolk Content of Mayonnaise

Dr. Wood, delegate from the UK, summarized the content of CX/MAS 88/12-Add.1. The WG agreed to recommend the endorsement of both methods described in this document as Type I methods.

6. Review of Methods of Analysis for Aflatoxins (CX/FAC 88/18-Add.1)

Owing to lack of time, the WG did not discuss the methods of analysis for aflatoxins. It suggested that documents prepared by the Secretariat should be sent to Governments for comment and methods of analysis for aflatoxins should be discussed at the next session.

#### 7. Abbreviations used in this Appendix

E = endorsed

TE = temporarily endorsed

NE = not endorsed

TBE = to be endorsed

EP = endorsement postponed

PART I - RECONSIDERATION OF METHODS ENDORSEMENT OF WHICH HAD BEEN POSTPONED

COMMODITY	PROVISION	METHOD	TYPE	STATUS	COMMENTS
CANNED MANGOES (CODEX STAN 159-1987) ALINORM 87/20	Syrup measurement 10-35° Brix (Refractometer method)	AOAC (1984) 31.011; or ISO 2173 (1978)	I	TE	1/
MANGO CHUTNEY (CODEX STAN	Total Soluble Solids (min. 50% m/m)	AOAC (1984) 31.011; or ISO 2173 (1978)	I	TE	1/
160-1987) ALINORM 87/20	Total Ash (max. 5% m/m) Ash insoluble in HCl (max. 0.5% m/m)	ISO 763-1982 (Ash insoluble in hydrochloric acid)	I	E	
HONEY (CODEX STAN 12-1981)	Hydroxymethylfurfural Content (max. 80 mg/kg)	ISO 7466-1986		NE	. 2/
SORGHUM FLOUR (at Step 5) ALINORM 87/29	Colour	Colorimetric method using the Kent Jones and Martin colour grader		NE	<u>3</u> /
VINEGAR (CODEX STAN 162-1987)	Residual alcohol (max. 0.5% v/v, except for 1% v/v in wine	AOAC (1984) 9.020-9.022 (specific gravity by pycnometer)	II	NE	<u>4</u> /
		OIV method, Recueil des méthodes inter- nationales d'analyses du vin, 1969, A-2-16	III	NE	4/
	Sulphur dioxide (max. 70 mg/kg)	OIV Method (iodometric titration), Recueil des méthodes intern- ationales d'analyses du vin, 1969, A-17.	II	NE	<u>5</u> /
	Iron (Fe) (max. 10 mg/kg)	IFJU method No. 15, 1964, (photometric method).	II	NE	<u>6</u> /
PROPOSED CODEX GENERAL METHOD (see also Appendix VII, ALINORM 89/23)	Tin (max. 150-250 mg/kg)	AOAC XIII (1984) 25.161-25.163 (AAS method)	III	NE	<u>7</u> /

COMMODITY	PROVISION	METHOD	TYPE	STATUS	COMMENTS
(Cont.) CONCENTRATED FRUIT JUICES AND FRUIT NECTARS PRESER- VED EXCLUSIVELY	Added Salt (in tomato juice; no maximum level specified)	AOAC (1984) 32.034 IFJU Method No. 37, 1968	III	E E	<u>16</u> /
BY PHYSICAL MEANS	Lead (max. 3 mg/kg)	AOAC (1980) 25.016- 26.067. IFJU Method No. 14, 1964 ISO Method 6633	III (	E NE TBE	<u>17</u> /
FOOD GRADE SALT (CODEX STAN 150-1985)	Sodium chloride (min. 97% on a dry matter basis)	Method described in Standard	I	E	
	Insoluble Matter	ISO 2479-1972	I	E	
	Sulphate	ISO 2480-1972	II	E	
	Halogens	ISO 2481-1973	II	E	
	Calcium and Magnesium	ISO 2482-1973	II	E	
	Potassium	ECSS/SC 183-1979 (volumetric method) or ECSS/SC 184- 1979 (AA Method)	III	E E	
	Loss on Drying	ISO-2483-1973	I	E	
	Copper (Cu) (max. 2 mg/kg)	ECSS/SC 144-1977	II	E	
	Arsenic (max. 0.5 mg/kg)	ECSS/SC 311-1982	II	E	
	Mercury (max. 0.1 mg/kg)	ECSS/SC 312-1982	II	E	
	Lead (max. 2 mg/kg)	ECSS/SC 313-1982	II	E	
	Cadmium	ECSS/SC 314-1982 (max. 0.5 mg/kg)	II	E	
PROCESSED TOMATO CONCENTRATES (CODEX STAN 57-1981)	Mineral impurities (max. 60 mg/kg based on diluted product of 8% solids)	AOAC (1984) 44.098	I	TE	18/

COMMODITY	PROVISION	METHOD	TYPE	STATUS	COMMENTS
COCOA BUTTERS (CODEX STAN 86-1981)	Copper (max. 0.4 mg/kg)	Colorimetric (diethyl- dithiocarbamate) AOAC (1984), 25.066-25.071	II	TE	8/
	Lead (max. 0.5 mg/kg)	Colorimetric Dithizone Determination Procedure after complete diges- tion, AOAC (1970) 25.053 (25.047-25.048)		NE	9/
CHOCOLATE (CODEX STAN 87-1981)	Copper (max. 15 mg/kg; 30 mg/kg)	Colorimetric (diethyl-dithiocarbamate) AOAC (1984), 25.066-25.071	II	E	
	Lead (max. 1 mg/kg; 2 mg/kg)	Colorimetric dithizone AOAC (1970) 25.053 (25.047, 25.048)		NE	9/
COCOA POWDERS (CODEX STAN 105-1981)	Copper (max. 50 mg/kg)	Colorimetric (Diethyldithiocarbamate) AOAC (1984), 25.066- 25.071	II	E	
	Lead (max. 2 mg/kg)	Colorimetric dithizone AQAC (1970) 25.053 (25.047-25.048)	11	NE	9/
COCOA BUTTER CONFECTIONERY (CODEX STAN 147-1985)	Percentage Cocoa Butter (min. 20% on dry matter)	AOAC (1980) 13.031- 13.033		EP	10/
GENERAL STAN- DARD FOR EDIBLE FATS AND OILS NOT COVERED BY INDIVIDUAL STANDARDS	<pre>Iron (max. 5 mg/kg; 1.5 mg/kg)</pre>	CAC/RM-1969 Determination of Iron Content	IV		11/
	Copper (max. 0.4 mg/kg; 0.1 mg/kg)	AOAC (1965) 24.023- 24.028	. II	TE	12/
	Lead (max. 0.1 mg/kg)	AOAC (1965) 24.053	II	NE	9/
EDIBLE LOW ERUCIC ACID RAPESEED OIL (CODEX STAN 123-1981)	Fatty acid composition (max. 5% m/m of the component fatty acids)	2.311	II	TE	13/
ALL CODEX	Ethanol (max. 3 g/kg)	IFJU Method No. 2 1968	IJ	NE	14/
STANDARDS FOR FRUIT JUICES,	Hydroxymethylfurfural	IFJU Method No. 12 1968	II	NE	15/

COMMODITY	PROVISION	METHOD	TYPE	STATUS	COMMENTS
TABLE OLIVES (CODEX STAN 66-1981)	Acidity of Brine (min. 0.4% m/m in terms of lactic acid)	Method described in standard	r	TE	<u>19</u> /
	pH of Brine (max. 4.0-4.5)	Method described in standard	II	TE	<u>19</u> /
RAISINS (CODEX STAN 67-1981)	Sulphur dioxide (max. 1500 mg/kg)	AQAC (1984) 20.126- 20.128	II	E	
DRIED APRICOTS (CODEX STAN 130-1981)	Sulphur dioxide (max. 2000 mg/kg)	AOAC (1984) 20.126- 20.128	II	E	
BOUILLONS AND CONSOMMES (CODEX STAN 117-1981)	Sodium chloride (max. 12.5 g/1)	Method 214 of AIIBP Official Collection of Methods of Analysis, (March 1975)		EP	<u>20</u> /
NATURAL MINERAL WATERS (CODEX STAN	Total Dissolved Solids	Method described in standard	I	TE	21/
108-1981)	Total Organic Matter	Permanganate method Handbuch der Leben- smittelchemie (Gesam- tred; J. Schormuller), Vol. VIII - Parts 1 and 2, Water and Air (S.W. Souci and K.E. Quentin) Springer- Verlag, 1969	I	TE	21/
GARI (CODEX STAN 151-1985)	Acidity (0.6 - 1% m/m as lactic acid)	AOAC (1975), 14.064- 065;	I	Е	22/
	Crude fibre	ISO 5498 (1981)	I	NE	23/

#### Footnotes

- The WG noted that the cited methods were not identical if dilution is necessary for the determination of darkly coloured products. The ISO method was temporarily endorsed as the more general method pending consideration of the above point by the Commodity Committee.
- The Secretariat is requested to bring to the attention of the Codex Alimentarius Commission that there is an equivalent alternative method (AOAC (1984) 31.153 XV) that does not use the carcinogenic reagent in the cited method and to determine whether this information should be provided to the Secretariat of the host country of the CCPFV to make a recommendation.
- $\underline{3}$ / Still awaiting additional support data on applicability of the method to the standard.

#### References

- Information requested of the Coordinating Committee for Europe on the origin and nature of residual alcohol was not provided. The WG also noted that there are modern gas chromatographic and enzymatic methods available for consideration.
- The WG noted that a modification of the classical Monier Williams method had recently been collaboratively studied within the AOAC and this version is not subject to interferences from volatile acids in vinegar and would therefore be suitable.
- 5/ Status retained; substitution of collaboratively studied methods suggested by the WG has not yet been considered by the Coordinating Committee for Europe.
- The WG recommended deleting the reference method and recommended replacing it with an improved method for tin in canned foods (AOAC 1st Supplement 1985, 25.A01) as a Type III method.
- Supporting data is needed from the Commodity Committee on method performance with cocoa butters at the standard level of 0.4 mg/kg as the WG is uncertain the method is applicable at this low level. The WG also recommends that the Commodity Committee consider adopting the IUPAC collaboratively studied graphite furnace AAS procedure as a Type III general method for Cu, Fe and Ni in oils and fats (Pure and Applied Chem., Vol. 60, No. 6. pp. 893-900, 1988).
- The WG believes the method is unreliable at the maximum level specified in the standard and therefore changed the status to "not endorsed". Dr. Lawrence of Canada will investigate to determine if there is an appropriate method to be recommended to the Commodity Committee and it was noted that a collaborative study of a method for lead in fats and oils has been completed by IUPAC.
- Continued in this status pending development of suitable methodology. The WG notes that sterol work and possible application of a UV light scattering approach may help address this problem.
- Method not collaboratively tested and should therefore be listed as Type IV. The WG also recommends that the Commodity Committee consider adopting the IUPAC collaboratively studied graphite furnace AAS procedure as a Type III general method for Cu, Fe, and Ni in oils and fats (Pure and Applied Chemistry, Vol. 60, No. 6 pp. 893-900, 1988).
- The WG is uncertain of applicability of the method at the 0.1 to 0.4 mg/kg standard levels and supporting data should be requested from the Commodity Committee.
- Mr. Kirk will contact the IUPAC Secretariat to obtain the needed collaborative study data relevant to Codex Standard 123-1981 for erucic acid.
- Status retained but OIV representative indicated that they will submit collaborative study data on a gas chromatographic method they are recommending. The IFJU is also requested to submit the results of their collaborative study and methodology recommendation to the Secretariat.
- The Codex Commission removed the specification for hydroxymethylfurfural and therefore no further action is needed.
- The WG recommended the endorsement of the recommendation of CCFJ to use the General Codex Method for chlorides (AOAC (1984) 32.034) as a Type II method and IFJU method No. 37-1968 as a Type III method.
- 17/ IFJU is requested to submit collaborative study data on ISO Method 6633.

#### References

- 18/ Request to provide the basis for selection of this method should be repeated to the Commodity Committee.
- 19/ Collaborative studies are needed for these methods.
- 20/ Recommendation to utilize the Codex General Method for Chlorides should be repeated to the Commodity Committee.
- 21/ Status was retained in light of the need for results of collaborative studies to be provided by the Commodity Committee.
- Following the CCMAS Session, the 8th Session of the Coordinating Committee for Africa clarified this provision as referring to product acidity rather than the acidity of extracted fat as determined by the AOAC method. The AOAC method was, therefore, withdrawn by the Coordinating Committee.
- 23/ Pending reconsideration by the Coordinating Committee for Africa.

PART II - Review of Methods of Analysis in Codex Standards elaborated by the Codex Committee on Nutrition and Foods for Special Dietary Uses

METHOD/TITLE	STANDARD REFERENCE	METHOD	TYPE	STATUS	COMMENTS
Silica (colloidal), Calcium silicate max. 1% m/m	CODEX STAN 53-1981 (salt Substitute)	AOAC XIV, 1984, 35.054	IV	*	
Iodine	_ " _	Not needed			
Fat in Foods containing starch, meat or vegetable products (quantity as declared on the label)	CODEX STAN 73, 74-1981 53,118-1981 (foods for infants and children, low- sodium foods, gluten-free foods)	CAC/RM 55-1976 (Vol. IX), n-Hexane extraction	I	Ε .	
Fat in Infant Foods not containing starch, meat or vegetable products (min. 3.3 g, max. 6 g/100Kcal)	CODEX STAN 72-1981 ALINORM 87/26, Appendix III (infant formula, follow-up formula)	Method B-2 of Code of Principles for Milk and Milk Products CAC/M 1-1973, Vol. XV of the Codex Alimentarius		Е	
Ash	CODEX STAN 53, 72-74, 118-1981; ALINORM 87/26, Appendix III	AOAC XIV, 1984, 7.009	I	E	
Crude protein	_ " _	Kjeldahl method for total nitrogen, text in Vol. IX of the Codex Alimentarius	I	E	
Loss on Drying	CODEX STAN 53, 72-74, 118-1981; ALINORM 87/26, Appendix III	AOAC XIV (1984) 7.003 Moisture Drying in Vacuo 95-100°C	I	TE	1/
Vitamin C (L-Ascorbic Acid) (min. 8 mg/100 Kcal)	CODEX STAN 72-1981 ALINORM 87/26, Appendix III	AOAC XIV, 1984, 43.069-43.075 (microfluorometric).	II	E	
, <u>, 2</u> , <u>2</u> ,	* E	AOAC XIV, 43.064- -43.068 (dichloro- indophenol)	III	E	2/

METHOD/TITLE	STANDARD REFERENCE	METHOD	TYPE	STATUS	COMMENTS
Thiamine (Vitamin B <sub>1</sub> ) (40 µg/100 kcal)	CODEX STAN 72-1981 ALINORM 87/26, Appendix III (infant formula and follow-up formula)	AOAC XIV, 1984, 43.024-43.030 (Fluorometric method)	II	E	
Riboflavin (Vitamin B <sub>2</sub> ) (60 µg/100Kcal)	CODEX STAN 72-1981 ALINORM 87/26, Appendix III (infant formula and follow-up formula)	AOAC XIV, 1984, 43.039-43.042 (Fluorometric method)	II ·	Е	
Nicotinamide for milk-based foods (250 µg/100 kcal)	CODEX STAN 72-1981 ALINORM 87/26, Appendix III (infant formula and follow-up formula)	AOAC XIV, 1984, 43.191-43.199 (Titrimetric/ Turbidimetric method)	II	Е	
Nicotinamide for foods not based on milk (250 µg/100 kcal)	_ " _	AOAC XIV, 1984, 43.048-43.050	II	E	
Vitamin B (35 پاع/100 kcal)	<b>- " -</b> .	AOAC XIV, 1984, 43.229-43.234	II	E	
Folic acid (4 µg/100 kcal)	CODEX STAN 72-1981 ALINORM 87/26, Appendix III (infant formula, follow-up formula)	AOAC XIV, 1984, 43.183-43.190	11	E	
Pantothenic acid (300 µg/100 kcal)	- " -	AOAC XIV, 1984, 43.200-43.208	II	E	
Vitamin B <sub>12</sub> (0.15 µg/100 kcal)	<b>- " -</b> .	AOAC XIV, 1984, 43.175-43.182	II	E	
Chloride (min. 55, max. 150mg/ 100 kcal)	- " -	Codex General Method App.IV, ALINORM 76/23	II	E	
Water Capacity of Containers	All standards	CAC/RM 46-1972, Vol. II of the Codex Codex Alimentarius Commission	I	Е	
Nitrogen	CODEX STAN 118-1981 (gluten-free foods	To be selected	-	-	<u>3</u> /

METHOD/TITLE	STANDARD REFERENCE	METHOD	TYPE	STATUS	COMMENTS
Calcium (min. 50 mg/100 kcal)	CODEX STAN 72-1981 ALINORM 87/26, Appendix III (infant formula,	AOAC XIV, 1984, 43.292-43.296	III	E	4/
	follow-up formula)				
Sodium (min. 20, max. 60 mg/kcal)	- " - )	IDF 119A/1987 or ISO 8070 (equivalent method); AOAC XIV, 1984,	III	E E	4/
Potassium (min. 80, max. 200 mg/kcal)	- " - )	43.292-43.296	<del></del>		<i>⊒</i> ⁄
Sodium Content Very low sodium: max. 40/100 g Low sodium: 120mg/100g	53-1981 (low sodium foods and	(proposed for Na, K,		NE	<u>5</u> /
Salt substitute		Ca, Mg)			
Potassium Content (not limited)	CODEX STAN 53-1981 (salt substitute)	_ " _			
Calcium Content (not limited)	<b>- " -</b>	AOAC (1984), 2.126- 2.130			*
Magnesium Content (max. 20% of cations K <sup>+</sup> Ca <sup>++</sup> and NH <sub>4</sub> )	- " -	AOAC (1984) 2.126- 2.130			*
Ammonium Content (max. 3% m/m)	- " -	AOAC (1984) 2.065			*
Phosphorus Content (max. 4% m/m)	_ " _	AOAC (1984) 43.292- 43.296	٠.		*
Protein Efficiency Ratio (PER)	CODEX STAN 72-1981; ALINORM 87/26, Appendix III (infant formula, follow-up formula)	AOAC XIV, 1984, 43.253-43.257	I	E	<u>6</u> /
Vitamin A (min. 75, max. 150 µg/ 100 kcal as retinol)	U	AOAC (1984) 43.A21	IV		<u>7</u> /
Vitamin D (min. 40, max. 80 i.u./100 kcal)	,     = " = .	AOAC (1984) 43.236- 43.249	IV		<u>7</u> /
Pantothenic acid (min. 300 µg/100 kcal)	<b>- "</b> -	USDA Handbook 97 or "The Analyst" for non-enriched foods: 89,1, 1964	IV		<u>7</u> /

METHOD/TITLE	STANDARD REFERENCE	METHOD	TYPE	STATUS	COMMENTS
Vitamin E (X-tocopherol compounds) 0.7.i.u./g linoleic acid or per g poly-unsaturated fatty acids	72-1981 ALINORM 87/26, Appendix III	AOAC XIV, 1984, 43.128-43.137	IV		10/
expressed as linoleic	follow-up foods) acid				
Phosphorus (min. 25 mg/100 kcal)		AOAC XIV, 1984, 22.040-22.042	II	E	<u>8</u> /
Copper (min. 60 µg/100 kcal)	<b></b>	AOAC XIV, 1984, 43.292-43.296			*
Manganese min. 5 μg/100 kcal)	- " -	AOAC XIV, 1984, 43.292-43.296			*
Zinc (min. 0.5 mg/100 kcal)	CODEX STAN 72-1981 ALINORM 87/26, Appendix III (infant formula, follow-up foods)	AQAC XIV, 1984, 43.292-43.296		. '	*
Magnesium (min. 6 mg/100 kcal)	_ # _	AOAC XIV, 1984, 43.292-43.296			*
Iron (0.15 mg/100 kcal)	- " -	AOAC XIV, 1984, 43.292-43.296			*
Choline (max. 3% m/m)	CODEX STAN 53-1981 (Salt substitute)	To be elaborated			
Choline (min. 7 mg/100 kcal)	CODEX STAN 72-1981; ALINORM 87/26, Appendix III (infant formula, follow-up formula)	Under elaboration by FDA/IFC		·	
Linoleate (in the form of glycerides) (min. 300 mg/100 kcal)	n _	IUPAC Standard Methods for the Analysis of Fats and Oils (to be published and AQAC XIV, 14.019	);	TBE	
		then 28.056-28.068 14.019 then 28.082- 28.085			
Crude Fibre	CODEX STAN 53, 72-74, 118-1981; Appendix III (low-sodium foods, gluten-free foods, foods for infants and children)	AOAC Enzymatic - gravimetric method modified version	I	TBE	<u>9</u> /

METHOD/TITLE	STANDARD REFERENCE	METHOD	TYPE	STATUS	COMMENTS
Vitamin K <sub>1</sub> (min. 4 µg/100 kcal)	CODEX STAN 72-1981 ALINORM 87/26, Appendix III (infant formula, follow-up formula)	To be elaborated			
Biotin (Vitamin H) (min. 1.5 µg/100 kcal)	_ " _	To be elaborated			
Iodine (min. 5 µg/100 kcal)	_ " _	To be elaborated			

#### Footnotes:

- \* Endorsement withdrawn
- The Commodity Committee should consider that high fructose products require drying at 70°C in vacuo and other food products can normally be done at 100°C without vacuo.
- 2/ Method should be used only for not highly coloured fruit juices and vitamin tablets.
- In view of the pending collaborative studies of an ELISA method for detecting gluten type proteins, the Commodity Committee may wish to consider a more specific method and standard.
- The WG endorsed this method as a Type III method because of the relative scarcity of the Inductively Coupled Plasma (ICP) equipment needed.
- The WG recommends that the Commodity Committee consider the use of an ion selective method (AQAC (1984) 43.271-43.274) for this purpose.
- The WG notes that two Type I methods must be identical and therefore the AOAC XIV, 1984, 43.258-43.267 method must be deleted as a Type I method.
- The Commodity Committee is awaiting results of a collaborative study in the matrix of interest.
- The WG endorsed the collaboratively studied method AOAC 43.B23-43.B28 (1986) and recommended it as a Type II method to the Commodity Committee.
- Reference to the modified collaborative version of the AOAC enzymatic-gravimetric method is Mitt. Gebiete Lebensm. Hyg. 79, 57-68 (1988) and the reference to the method is Schweiz. Lebensmittelbuch Kap. 22, Method 22/8.2 (1987). The WG also suggests the Commodity Committee utilize the term fiber rather than crude fiber when referring to this method.
  - 10/ To be studied collaboratively with reference to the matrix.

COMMODITY	PROVISION	METHOD	TYPE	STATUS	COMMENTS
PROPOSED CODEX GENERAL METHOD (see also App.VI of ALINORM 89/23		NMKL Quercetin method No. 115 (1985)	IV	-	1/
EDIBLE GRAPE- SEED OIL (CODEX STAN 127-1981)	Erythrodiol content (min. 3% of the beta-sitosterol content)	IUPAC Standard Method of Analysis for Oils, Fats and Derivatives, 7th Ed., 1987, 2.431	II	E	
DRAFT STANDARDS FOR SPECIFIED VEGETABLE FAT	Acid value (min. 0.6 mg KOH/g) (max. 0.8 mg KOH/g)	IUPAC, 6th Ed., 1979 2.201 or ISO 660	I	E	2/
PRODUCT (at Step 8) (App.II, ALINORM 87/17) AND VEGETABLE FAT PRODUCTS (at Step 8) (App. III, ALINORM 87/17)	Peroxide value (max. 10 mg peroxide oxygen/kg)	IUPAC, 6th Ed., 1979 2.501 - Standard Method for the Analysis of Oils, Fats and Derivatives; or ISO 3960 - (equivalent methods)	I	Е	
	Slip point (31-44°C)	AOCS Official Method cc 3-25 (1983)	I	TBE	3/
	Vitamin A  µg retinol (vit A - alcohol) per kg  product (no level given)	AOAC (1984) 43.001-43.007	II	Е	<u>4</u> /
	Vitamin D µg Vit. C/kg product (no level given)	AOAC (1984) 43.236-43.249	II	E	<u>5</u> /
	Vitamin E mg of each tocopherol per kg of product (no level given)	IUPAC (1981) method (IUPAC standard methods for the Analysis of Oils, Fats and Derivates, 6th Ed., 1st Supplement, Part 4, 1981, 2.404)	II	E	<u>5</u> /
	Volatile matter (max. 0.2% m/m)	IUPAC (1979) method (IUPAC Standard Method for the Analysis of Oils, Fats and Derivatives, 6th Ed., 1979, 2.601 or ISO 662-1980 (equivalent methods)	I	E	<u>6</u> /

COMMODITY	PROVISION	METHOD	TYPE	STATUS	COMMENTS
Idem	Insoluble Impurities (max. 0.05% m/m)	IUPAC (1979) method (IUPAC Standard Methods for the Analysis of Oils, Fats and Derivatives, 6th Ed., 1979, 2.604 or ISO 663-1981 (Equivalent methods)	I	Е	
	Soap Content (max. 0.005% m/m)	FAO/WHO Codex Alimen- tarius Method (FAO/ WHO Methods of Analysis for Edible Oils and Fats, CAC/RM 13-1969, Deter- mination of Soap Content)	İ	<b>E</b>	
	Iron (Fe) (max. 1.5 mg/kg)	AOCS Method Ca 18-79	II	Ē	
	Copper (Cu) (max. 0.1 mg/kg) Lead (Pb) (max. 0.1 mg/kg)	AOCS Method Ca 18-79	II II	E TBE	<u>7</u> /
	Arsenic (As) (max. 0.1 mg/kg)	AOAC, 1984, 14th Ed., 25.048-25.049	II	E	
MARGARINE (CODEX STAN 32-1981)	Milk Fat Content	CAC/RM 15-1969 Determination of Milk Fat	I	E	<u>8</u> /
MINARINE (CODEX STAN 135-1981)	Milk Fat Content	CAC/RM 15-1969 Determination of Milk Fat	I	E	<u>8</u> /
PROPOSED DRAFT STANDARD FOR DURUM WHEAT FLOWER AND	Moisture (max. 15%)	ISO 712-1985 (air oven) or ICC 110/1 (equivalent method)	I	E	
SEMOLINA (at Step 3) (App.V, ALINORM 87/29)	Particle Size (Granularity)	AOAC 14th Ed. (1984) - Cereal Adjuncts - Sorting of Corn Grits, Sieving Method 10.162- 10.163	I	E	
	Ash: Semolina (max. 0.8% - 14% moisture basis, 0.93% - dry basis)	AOAC 14th Ed. (1984) - Cereal Foods - Direct Method, 14.006 (550°C to constant weight);	1	E	
	Ash: Durum wheat flour (max. 1.4% - moisture basis, 1.63% - dry basis)		1	E	

COMMODITY	PROVISION	METHOD	TYPE	STATUS	COMMENTS
	Fat Acidity	AACC 02-01A - Fat Acidity - General Method.	I	E	. •
	Protein (N x 5.7) (min. 11% (14% moisture basis), 12.8% (dry basis))	ICC 105/1 - Method for the Determination of Crude Protein in Cereals and Cereal Products for Food and for Feed. Selenium/copper catalyst.	II	E	

#### Footnotes:

- The USSR may conduct a collaborative study at the levels of interest (150-250 mg/kg) in the future. The NMKL collaborative study was conducted at levels greatly below those at standards of interest but the method is available even though it is not endorsed for use at the 150-250 mg/kg level.
- $\frac{2}{}$  ISO representative stated that ISO 660-1988 method is identical to the IUPAC method.
- 3/ The WG recommends that the Commodity Committee reevaluate the proposed method in comparison to the recently studied ISO procedure 6321 and make a new recommendation as to which method is preferable.
- The Secretariat was requested to inform the AOAC that the CCMAS had endorsed this method and recommended the removal of the surplus method designation until more modern HPLC methods have been collaboratively studied.
- More modern methods should be considered by the Commodity Committee once they have been colaboratively tested.
- 6/ The WG has no objection to changing of the temperature to 103<sup>O</sup>C for determination of volatile matter.
- The WG recommends that the Commodity Committee consider the recently collaboratively studied IUPAC/ISO method for Cu, Fe, Ni and Pb (soon to be published) and make a new recommendation for a Type II reference method as the WG is uncertain about whether these methods will really determine the specification level.
- The WG recommends that the Commodity Committee revise and repropose the IUPAC method for butyric acid through the normal process. It was noted that the Codex Standard does not cover the level of milk fat and the Committee should consider this in their revision. Presumably this method is required by national legislation.



#### INTERNATIONAL RECOMMENDATION

#### NET CONTENT IN PACKAGES

#### 1. Introduction

1.1. This Recommendation specifies legal metrology requirements for labelled packaged commodities with constant nominal content and subject to international trade. It also presents sampling plans which may be utilized in net content verification of goods declared in units of mass or volume.

#### Notes

- a) for other measurable quantities (length, area, number...) analogous procedures may be considered, which however are not the subject of this Recommendation.
- b) the methods outlined in the Recommendation while suitable for legal metrology testing are inadequate for use in manufacturing quality control.
- 1.2. The term "net" or "net content" means the quantity of the commodity in the package exclusive of wrappers and any other material packed with such commodity. In this Recommandation this term is designated by the symbol  $Q_n$ .
- 1.3. The requirements for the labelling of packaged commodities are given in the OIML International Recommendation  $N^{\circ}$ .. "Information on package labels".
- 1.4. The statistical language follows the vocabulary of ISO 3534.

#### 2. Metrological requirements for packages

The following requirements shall be fulfilled when the goods are ready for sale at the point-of-pack or, where applicable, at the point-of-import.

#### 2.1. Average content

The average net content conveyed by any lot of packaged goods available for inspection shall equal or exceed the net content as declared on the package.

If the average net content in a lot is estimated by sampling, the criteria in points A.2.1 and A.3.1 shall be satisfied.

Note:A "lot" is assumed to comprise a sufficient number of units to represent the manufacturer's packing practice. As an example, in the sampling plan given in the Appendix B, point B.1, a minimum lot size of 150 units is demanded.

#### 2.2. Contents in individual packages

The declaration of net content shall accurately reveal the quantity of the commodity meant to be in the package; deficiencies from the stated quantity shall be permitted when caused by fluctuations in the filling process; however, packages underfilled shall be deemed to be non-conforming when their deficiency exceeds a given tolerable deficiency T (see B.1).

If the presence of non-conforming packages in a lot is checked by sampling, the criteria in points A.2.2 and A.3.2 shall be satisfied.

#### 2.3. Storage effects

Changes which are caused by ordinary and customary exposure to conditions which normally occur in storage and retail distribution shall be recognized by the inspection authority, in the evaluation of both average and individual contents.

#### 3. Accuracy of net content measurements

The determination of net contents shall be made within the limits of uncertainty of  $\pm$  0.2 T.

#### 4. Judgement of lots

The Appendixes present information on the statistical tests. The purpose is to permit the use of several different sampling plans while still giving packers information about applicable limits.

#### APPENDIX A

#### STATISTICAL TESTS General rules

#### A.1. Criteria

The tests to which the lots are subjected shall lead to acceptance or rejection of those lots depending on:

- the average net content  $\mu$  of the packages in the lot,
- the percentage p of non-conforming packages in the lot (which convey a content less than  $Q_n T$ ).

A lot is accepted if it satisfies the conditions fixed for both these parameters.

- A.2. Significance level of the tests
- A.2.1. With respect to average : the tests shall have a significance level  $\alpha_{\mu}$  of  $\alpha_{\mu} \leq 0.5 \ \text{% for } \mu = Q_{\pi}$

that is to say, the probability of rejecting a lot filled in average to  $\mu = Q_n$  shall not exceed 0.5 % if the percentage of non-conforming packages in this lot does not influence the result.

A.2.2. With respect to percentage of non-conforming packages, the tests shall have a significance level  $\alpha_{\rm p}$  of

 $\alpha_p \le 1$  % for p = 1 % that is to say, the probability of rejecting a lot containing 1% of non-conforming packages shall not exceed 1 % if the lot average does not influence the result.

- A.3. Type II risk
- A.3.1. With respect to the average, the tests shall, in at least 90 % of the cases, detect lots for which the filling is less than  $(Q_n 0.74 \sigma)$  where  $\sigma$  is the standard deviation of the net contents in the lot.
- A.3.2. With respect to percentage of non-conforming packages, lots containing 16 % of non-conforming packages shall be detected by the tests in at least 90 % of cases.
- A.4. Examples of sampling plans

Appendix B describes in point B.1 a sampling plan which is an economic combination of an average test and an attributive test fulfilling the conditions of points A.2 and A.3.

#### APPENDIX B

#### SAMPLING PLANS Recommended examples

#### B.1. OIML sampling plan for general use

This plan applies to packaged goods with contents declared in units of mass or volume for lots of at least 150 packages (on production lines, a lot is the output of 1 hour).

Sample size : 32 packages

Condition for the average :  $\bar{x} \ge Q_n - 0.485 \text{ s}$ 

Condition for non-conforming packages: at most 2 in the sample.

The average test follows ISO 2854 (comparison of a mean with a given value) with  $t_{0.995}(31)$  /  $\sqrt{32}$  = 0.485 (one-sided case).

The attributive test for non-conforming packages follows ISO 2859, code letter G, normal inspection, single sampling.

The acceptable individual deficiencies are given in the Table I below.

Table I
Acceptable individual deficiencies

Nominal net content $Q_n$ g or ml			Tolerable define percent of $Q_n$	
5	to	50	9	-
_			<u>-</u>	4.5
50	to	100		1.0
100	to	200	4.5	-
200	to	300	<del>-</del>	9
300	to	500	<b>.</b> 3	-
500	to	1 000	-	15 .
1 000	to	10 000	1.5	<del></del>
10 000	to	15 000	· –	150
15 000	to	25 000	1.0	-

These T-values are to be rounded up to the next tenth of g or ml for  $Q_n$  less than or equal to 1 000 g or ml and to the next whole g or ml for  $Q_n$  higher than 1 000 g or ml.

## B.2. Sampling plan for large lots

Lots of more than 4 000 packages may sometimes be divided and the parts treated as recommended in B.1. If this is not suitable, it may be advantageous to use a larger sample size than in the plan B.1. The alternative suggested is:

Sample size : 80 packages

Condition for the average :  $\bar{x} \ge Q_n - 0.295 s$ 

Condition for non-conforming units : at most 5 in sample

The average test follows ISO 2854 (comparison of a mean with a given value) with  $t_{0.995}(79)$  /  $\sqrt{80}$  ≈ 0.295 (one-sided case).

The attributive test for non-conforming packages follows ISO 2859, code letter J, normal inspection, single sampling.

The tolerable deficiencies (T) are the same as in the OIML plan B.1.

- B.3. General remarks on sampling
- B.3.1. Lots are assumed to be homogeneous as long as there is no indication to the contrary.
- B.3.2. If the lot is taken from a production line, it shall consist of all packages which are not rejected by the production's checking system and which are produced during a well defined lapse of time, such as one hour or the time necessary to produce 10 000 units at the actual pace of that line. Care must be taken to prevent any corrective actions other than those normally taken.
- B.3.3. In a store the lot considered must first be well defined.
- B.3.4. If a lot is larger than 10 000 units it should if possible be divided.
- B.3.5. Sampling should be done in such a way that all combinations of units in the defined lot have the same a priori probability of being chosen (Simple random sampling: ISO 3534 point 3.6).
- B.3.6. In a large warehouse random sampling according to B.3.5 may not be practical. In such a situation, other sample selection procedures can be applied if accepted by the person or party responsible for the goods.

# PROBLEMS ARISING FROM THE CLASSIFICATION OF ANALYTICAL METHODS SPECIFIED IN CODEX ALIMENTARIUS STANDARDS\*

#### 1. Background

The Codex Committee on Methods of Analysis and Sampling (CCMAS) has, in earlier sessions, clarified the purpose and definition of Codex methods of analysis. Its recommendations have resulted in the revised General Principles for the Establishment of Codex Methods of Analysis which appear in the 6th Edition of the Procedural Manual, and includes definitions for four types of methods of analysis, as follows:

Type I - Defining Methods - methods which determine a value that can only be arrived at in terms of the method per se and which can serve for calibration purposes.

Type II - Reference Methods - methods which are the single designated reference methods where Type I methods do not apply. The methods should be selected from Type III methods and are recommended for use in cases of dispute and for calibration purposes.

Type III - Alternative Approved Methods - methods which meet the criteria required by CCMAS for methods that may be used for control, inspection or regulatory purposes.

Type IV - Tentative Methods - methods which have been used traditionally or have been recently introduced, but for which the criteria required by CCMAS have not yet been determined.

The Codex Alimentarius Commission (CAC) decided in 1985 that Type I methods, because of their defining nature, should be subject to acceptance along with the relevant values so defined in the Standards. It was also decided that Type II methods should be obligatory only for cases of dispute concerning the results of analysis, whereas Type III methods were only advisory, i.e. recommended as suitable for general use. Type IV methods, if listed, were only tentative until they had been fully evaluated and endorsed by CCMAS as a Type III method.

Some Codex Commodity Committees have since reviewed the classification of methods in their Standards and at its 1986 Session CCMAS completed a full review of the classification of Codex methods of analysis as a part of its methods endorsement programme, with the help of a comprehensive listing prepared by Australia (see ALINORM 87/23).

Difficulties and differences of opinion in classifying some Codex methods, especially with regard to Types I and II, have however arisen, and this paper has been prepared to review the definitions and to outline some areas of debate.

<sup>\*</sup> Paper CX/MAS 88/9 prepared by the U.K. and distributed during the 16th Session of the CCMAS.

## 2. What is a Type I and Type II Method?

According to the definition in the Procedural Manual, a Type I method is a defining method which, when carried out exactly by the prescribed procedure, supplies and defines the analytical value or attribute given in the Standard. This means that the Standard is entirely dependent on the method for that particular value or attribute. No other method can be used. The implication is therefore that Type I methods are either empirical, measuring chemically or physically indefinite quality or compositional criteria, or very selective quantitative methods of analysis which measure a basic physical property or chemical constituent unobtainable by any other method or calculation. In cases of dispute involving such criteria, the Type I method as prescribed must also be used for reference or arbitration purposes.

Only Type II methods are defined as reference methods, presumably to be used where the dispute does not involve criteria obtained by Type I methods. The definition of Type II methods could be clearer on this point. If a reference method is one which is designated to settle disputes, does not this imply that it is also a defining method? Type II methods are also defined as being selected where possible from a list of suitable evaluated alternative Type III methods as most suitable for use in cases of dispute. These factors imply that Type II (and III) methods are not empirical but are absolute methods which determine actual chemical or physical values or attributes.

#### Calibrations

The current definitions of Type I and Type II methods both include a phrase that they can be used for calibration purposes. If however a Type I method is a unique defining method with no alternatives (and which therefore must be carried out to test foods and other materials against the criteria in Codex Standards), for what calibration purposes can it be used?

Is the phrase "for calibrating purposes" confusing as used for both Type I and Type II methods? Is clarification needed?

## 4. Systematic Bias

If a Type I method, when carried out exactly as prescribed, is incapable of producing a wrong result (since it is a defining method), does it follow that it is also incapable of showing systematic bias or error? This may be true for empirical methods but does it apply to non-empirical methods, some of which may be Type I only because they incorporate a criterion-defining factor in their calculation?

If a Type II method for a particular chemical component is such that no systematic bias is possible, can it be regarded as Type I method?

### 5. Examples of Problems in Classifications

#### 5.1 Loss on Drying

For Type I methods the Procedural Manual gives four examples, viz Howard Mould count, Reichert Meissl value, loss on drying and salt in brine by density.

The first two are clearly empirical methods as is also the salt in brine method on the assumption that the brine is not solely a solution of sodium chloride in water. "Loss on drying" however is not an empirical method, although it would be if the standard criterion was for moisture content, which is not specifically measured by a loss on drying technique. Loss on drying by specified times and temperatures are widely used in national and international reference methods. Moreoever different times and temperatures can produce virtually identical results, which seems to contradict the definition of a Type I method.

#### 5.2 Fat Content

It is well known that the composition and quantity of oil or fat extracted from a foodstuff will depend on the method of extraction and the solvents used. This is especially so for foodstuffs which are relatively rich in phospholipids and other bound lipids. Yet some Codex Standards list more than one extraction method. CCMAS has tried to satisfy the definition of a Type I method by recommending the selection for the standard on edible ices, the Rose Gottlieb method as Type I, and relegating the alternative method (Weibull) to a Type III, to be used routinely with calibration against the Rose Gottlieb method. Is this recommendation realistic or possible? If one method needs calibration by another, what method was used to define the standard? If either method was originally used to monitor the standard, according to the definition how can only one of them be the Type I method? If both are Type I empirical methods, it is not feasible to calibrate one with the other. Why relegate the most time-consuming method to a Type II, recommended for routine work?

The Weibull method is an ISO reference total lipid method. Can it be regarded on scientific grounds as Type II where there is a "fat" criterion in the standard and the method is listed?

#### 5.3 Acid Value/Free Fatty Acids

Both these values are given as criteria in the Codex Standard for olive oils. Both are determined by the same method and technique but the titration result is expressed differently. Free fatty acid content is empirically calculated as a single fatty acid (oleic) and would therefore be a defining Type I. CCMAS has classified Acid Value also as Type I although it appears to be an absolute Type II method giving acidity as mg KOH/g.

If an analytical criterion can be specified in two equivalent forms, does it make sense to have two different method types? Is it necessary to differentiate methods when they only differ in a calculation or expression?

#### 5.4 Sodium Chloride or Salt Content

In the Procedure Manual, the example given of a Type II method is the potentiometric method for halide, and for a Type III the Mohr and Volhard methods for chloride. These seem to be good examples only if the Codex Standards contain analytical criteria for chloride content. However many of the Standards specify salt or sodium chloride which is calculated from the result of the Type II or III method on the assumption that all the chloride in the food product is from sodium chloride. Since this is in fact an empirical assumption, should not the methods be Type I? Is the significance of the calculation in these methods being disregarded as defining the result?

### 5.5 <u>pH</u>

The CCMAS has classified the measurement of pH as Type I perhaps because the glass electrode and the calibration using buffer solutions is specified and no other procedure is possible. However the method actually measures hydrogen concentration, and Type II spectrophotometric methods for the measurement of chemical constituents also require similar specification and calibration. Is the measurement of pH any different from the measurement of refractive index which is classified as Type II?

#### 5.6 Protein/Nitrogen Content

The CCMAS has classified the Kjeldah method, where the result is expressed as nitrogen content, as Type II. When the result is required to be expressed as protein content, the same method is deemed to be a Type I or Type II method depending on which nitrogen conversion factor is used. (Type II if a specific factor or Type I if a general factor). Nevertheless for many foods, the general 6.25 nitrogen-to-protein factor gives a reasonably accurate estimate of the protein content. On the other hand, if the specification should require a protein content of "not less than x per cent (N x 6.25)", is the Kjeldahl method used Type I or Type II?

#### CODEX GENERAL METHODS FOR METALLIC CONTAMINANTS IN FOODS

#### Introduction

The methods of analysis listed below have been adopted by the Codex Alimentarius Commission as general methods, except for the proposed draft alternative approved method for copper, which is at Step 3 of the Codex Procedure. The Codex Committeee on Methods of Analysis and Sampling agreed that the general methods should be reviewed at the 17th Session on the basis of comments and information received (see paras. 70-71).

It should be noted that only one Codex Reference Method will be selected as such methods are intended to be accepted by Governments for use in situations of dispute. On the other hand, any number of Codex Alternative Methods may be selected.

The following documents should be consulted:

- (a) General Principles for the Establishment of Codex Methods of Analysis (Procedural Manual of the Codex Alimentarius Commission, 6th Ed.);
- (b) Information required by the CCMAS for consideration of methods of analysis (Annex 1 to Appendix II, ALINORM 85/23)

#### CODEX GENERAL METHODS FOR METALLIC CONTAMINANTS IN FOODS

#### COPPER (Cu)

Codex Reference Method (Type II)
AOAC XIII, 1980, 25.044-25.048 (atomic absorption method)

Proposed Draft Codex Alternative Approved Method (Type III) (at Step 3) AOAC XIV, 1984, 25.066-25.071 (diethyldithiocarbamate method) (Step 3)

#### ARSENIC (As)

Codex Reference Method (Type II)
AOAC, XIII, 1980, 25.012-25.013 (silverdiethyldithiocarbamate method)

Codex Alternative Approved Methods (Type III)

- AOAC, XIII, 1980, 25.010-25.011 (colorimetric, molybdenum blue method) (for levels down to 1 mg/kg)
- AOAC, XIII, 1980, 1st Supplement 25.A01-25.A05 (hydride generation atomic absorption method) (to be calibrated against a reference method if other than Perkin Elmer equipment is used).

#### CADMIUM (Cd)

Codex Reference Method (Type II)
AOAC, XIII, 1980, 25.026-25.030 (atomic absorption method)

Codex Alternative Approved Method (Type III)
AOAC, XIII, 1980, 1st Supplement, 25.A01-25.A04 (anodic stripping voltametry method) (for levels down to 0.1 mg/kg)

#### MERCURY (Hg)

Codex Reference Method (Type II)
AOAC, XIII, 1980, 25.110-25.116 (for fish and sea food)

Codex Alternative Approved Method (Type III)
None

#### LEAD (Pb)

Codex Reference Method (Type II) AOAC, XIII, 1980, 25.061-25.067 (for levels down to 0.3 mg/kg)

Codex Alternative Approved Methods (Type III) AOAC, XIII, 1980, 1st Supplement, 25.A03-25.A04 (closed digestion anodic stripping method) (for levels up to 1 mg/kg)

#### ZINC (Zn)

Codex Reference Method (Type II)
AOAC, XIII, 1980, 25.150-25.153 (atomic absorption method)

Codex Alternative Approved Method (Type III) AOAC, XIII, 1980, 1st Supplement, 25.A03-25.A05 (closed digestion atomic absorption method) (for levels up to 15 mg/kg)

#### TIN (Sn)

Codex Reference Method (Type II)

AOAC, XIII, 1980, 25.136-25.138 (at levels of 10 mg/kg)
(withdrawal proposed, see Appendix IV, Part I, footnote 7, ALINORM 89/23)

Codex Alternative Approved Method (Type III)

AOAC 1st Supplement 1985, 25.A01
(See Appendix IV, Part I, footnote 7, and Part III, footnote 1, ALINORM 89/23)

#### REPORT OF THE SEVENTH INTER-AGENCY MEETING (IAM)

#### Budapest 10-11 November 1988

#### TABLE OF CONTENTS

	Para
Review of Membership of IAM	6 7 11 17–27 28–31 32–35
- cocoa products - milk products and edible ices - fats and oils - foods for special dietary uses - processed fruits and vegetables - processed meat and poultry products - sugars and starch hydrolysis products - cereals, pulses and legumes - microbiological methods - mineral waters - contaminants - food additives - wines and spirits	37-38 39-42 43-44 45-47 48 49 50-52 53-56 57-60 61 62-63 64-65 66-69
Nutritional labelling	70-71 71-72 73-76 77-80 81-82 83-84

#### OPENING OF THE MEETING

- 1. The Meeting was opened by Dr. K. Sütö, Vice-President of the Hungarian Office for Standardization and President of the Hungarian National Codex Committee. Dr. Sütö extended a warm welcome to the Representatives of the various International Organizations (see Annex 1) and stressed the importance of international standardization as one of the means for fostering cooperation between different nations. He also mentioned the role of the Inter-Agency Meeting (IAM) as an advisory body which contributes to the success of the Codex Committee on Methods of Analysis and Sampling.
- 2. Dr. Sütő then introduced Mrs. E. Nagy who had succeeded Mr. K. Kismárton as Secretary of ISO/TC 34 "Agricultural food products" and Mrs. E. Karsay, the new Secretary of ISO/TC 34/SC4 "Cereals and pulses".

#### ELECTION OF CHAIRMAN

3. Upon the suggestion of Dr. Sütö, Mr. G. Castan, Chief of Priority Programmes of AFNOR was elected Chairman.

4. After thanking for the confidence, Mr. G. Castan drew attention to the aim of the IAM which is to assist the CCMAS in its efforts to standardize methods of analysis and sampling that are suitable for the purpose of the Codex.

#### ADOPTION OF THE AGENDA

5. The Representative of IDF proposed an additional agenda item under which the work of the Codex Committee on Food Labelling should be discussed. This was agreed and the agenda was adopted, subject to inclusion of a new item 10.17 "Other activities of interest to the Inter-Agency Meeting".

#### REVIEW OF MEMBERSHIP OF THE INTER-AGENCY MEETING

6. The Meeting noted with regret that ARSO, ASMO, CCE, IFJU and IUMS had not responded to the invitation to send representatives to the IAM. In view of the possible interest of these Organizations in the work of the IAM, however, it was agreed to retain their addresses on the mailing list.

# ACTION TAKEN BY THE CODEX SECRETARIAT IN RESPONSE TO THE RESULTS OF THE SIXTH INTER-AGENCY MEETING

- 7. The Representative of the Codex Secretariat informed the Meeting that the following action had been taken by the Codex Secretariat:
  - The report of the Sixth Inter-Agency Meeting had been edited and published as Appendix IV of the Report of the Fifteenth Session of CCMAS (document ALINORM 87/23);
  - Action had been taken that relates to CCMAS and the CAC itself;
  - A Representative of CAC had attended the IUPAC/AQAC/ISO Workshop on the Harmonization of Collaborative Analytical Studies, held in Geneva on 4 and 5 May 1987.
- 8. The Representative of the Codex Secretariat also drew attention to document CX/MAS 88/14 which would be considered at the Sixteenth Session of CCMAS. This document contains a list of methods of analysis required for Codex Standards and which need to be developed and/or validated. Some of these methods are totally missing, some others would have to be validated. It will be the task of CCMAS to see whether appropriate methods exist.
- 9. In reply to the suggestion by the Representative of the Codex Secretariat that it may no longer be possible in future to include the IAM report as an appendix in the report of CCMAS, the Secretary expressed the hope that the Codex Secretariat would still be able to assist the IAM Secretariat in the editing of the final version of the IAM report even if this would have to be circulated as a separate Codex document.
- 10. The Meeting then discussed the possibility of holding future Inter-Agency Meetings after the Sessions of CCMAS. As an important part of the discussions at CCMAS Sessions relates to the work of IAM, however, it was decided not to change the current practice of holding the Inter-Agency Meetings before the Sessions of the CCMAS.

# SUGGESTIONS FOR IMPROVING INTERNATIONAL COOPERATION IN THE FIELD OF STANDARDIZED METHODS OF ANALYSIS

- 11. The Representatives of IDF and NMKL noted with satisfaction that the recommendations with respect to cooperation in the exchange of information on collaborative studies as adopted at the previous IAM had been implemented by several Organizations. Nethertheless, only AOAC, IDF and NMKL seemed to exchange such type of information on a regular basis.
- 12. The Meeting recognized that bilateral contacts existed between various Organizations which are not necessarily known to all other interested Organizations.

- 13. The Representative of the Codex Secretariat noted the role of the IAM as a type of catalyst which may encourage increased cooperation between interested Organizations.
- 14. The Representative of ICUMSA felt that the relevant information should be centrally collected and distributed to all interested Organizations. This would also require that the information be presented in accordance with the recommendations agreed upon by IAM. The Meeting therefore accepted the offer of AOAC to collect and circulate information on collaborative studies planned or carried out by the interested Organizations. This information should be sent to the European Representative of AOAC, at the following address:

Mrs. Margreet Lauwaars Langhoven 12 NL-6721 SG BENNEKOM The Netherlands

- 15. The Secretary was requested to inform all interested Organizations of this decision and to circulate the recommendations adopted at the previous IAM, amending item (f) of these recommendations as appropriate.
- 16. In regard to item (h) of the recommendations, the Representative of the Codex Secretariat stressed the wish of the IAM to be informed of all collaborative studies. The CCMAS should be invited to specify the exact matrices or the parameters of the methods required and to identify the need for its request (i.e. method lacking, new method or method to be validated).

#### COLLABORATIVE TESTING OF METHODS OF ANALYSIS

17. Under this agenda item, the Meeting considered the comments and suggestions of various Organizations interested in the subject of collaborative analytical studies.

#### STATUS OF RELEVANT WORK WITHIN ISO/TC 69 "APPLICATIONS OF STATISTICAL METHODS"

- 18. In the absence of Mr. E. Nouat (ISO), the Secretary gave a brief account of the work of ISO/TC69. He emphasized the importance of ISO 5725:1986 "Precision of test methods Determination of repeatability and reproducibility for a standard test method by interlaboratory tests" to the work of ISO Committees and to other Organizations. This Standard is being revised and will eventually be superseded by a new edition which will also take into account the comments received from other Organizations such as AOAC, IDF and IUPAC.
- 19. The Representative AOAC said that ISO 5725:1986 was a broad standard which was primarily oriented towards the needs of statisticians and which did not necessarily cover the practical needs of the analytical chemist in the organization of collaborative studies.

# STATUS OF JOINT WORK BY AQAC, IUPAC AND ISO TOWARDS A HARMONIZED PROTOCOL FOR COLLABORATIVE STUDIES

- 20. The Representative of AOAC informed the Meeting that following the AOAC/IUPAC/ISO Workshop held in Geneva in May 1987, a final draft of the Protocol for the design, conduct and interpretation of collaborative studies had been published in July 1988. He invited interested Organizations to transmit their comments by 1 December 1988. A further meeting of the IUPAC Working Group on Harmonization will be held in Washington D.C. 17-20 April 1989 on Harmonization of Quality Schemes in Chemical Analysis and on the Adoption and Presentation of Analytical Methods Standardized by Collaborative
- 21. The Representative of IDF mentioned that IDF Standard 135:1985 on the subject of collaborative studies will shortly be superseded by a new edition which takes into account the results achieved by the Harmonization Group. He also informed the Meeting that an IDF Group of Experts was preparing a document on the design and organization of collaborative studies for microbiological methods.

### VALIDATIONS OF METHOD OF ANALYSIS

- 22. The Representative of ICUMSA informed the meeting of the work carried out in the United Kingdom on the validation of methods which resulted in the publication of a draft protocol. This draft protocol can be applied to collaborative studies by which no discrete answers are obtained. The responsible group also considers quality control procedures. Furthermore, the group organizes cooperative trials in which each participant uses his own method rather than a specific method. This activity is of interest in view of the EEC efforts towards the internal market in 1992.
- 23. As a result of the ensuing discussion, the Representatives of all interested Organizations were invited to exchange any information available with respect to the validation of methods. Reference was also made to IDF Standard 207:1986 on the use of reference materials and the catalogue of the CEC Bureau of Reference Materials. The representative of ISO informed the meeting about a new approach for validation, viz. by application of criteria for proving the presence of an analyte in a matrix instead of by meticulously described methods. Such criteria have been stated for a number of analytical methods, and are included in a draft EEC document (VI/1591/88-EW) on "Criteria for reference methods of analysis for residues".

#### PRACTICAL COOPERATION IN COLLABORATIVE TESTING

- 24. The Representative of IDF informed the Meeting of a document on the interlaboratory studies carried out by the Joint IDF/ISO/AOAC Group of Experts. He mentioned that other Organizations were welcome to participate in the respective work.
- 25. The Representative of AOAC stressed the importance of quality control of methods in the study of methods on the basis of a practical example.
- 26. The Representatives of NMKL and IFG reported on relevant activities of their Organizations. In the same context, the work of ISO/TC93 "Starch (including derivatives and by-products)" on starch hydrolysis products was mentioned.
- 27. The Chairman invited all Organizations to send any relevant information to the IAM Secretariat so that the existing cooperation could be further improved. The Codex Secretariat was invited to help to identify priorities and to clarify the real needs of the CAC as far as suitable methods are concerned.

#### SAMPLING METHODS FOR FOOD PRODUCTS

- 28. The Representative of the Codex Secretariat informed the Meeting that an internationally acceptable standard approach to sampling would still have to be defined. The CAC should decide what is required to facilitate the international trade of food products. He also referred to the discussions on administrative sampling procedures by the CCMAS and a document covering sampling plans for net content which has been prepared by OIML.
- 29. The Representative of the Codex Secretariat expressed the hope that CCMAS would be able to identify the role of international organizations in the development of internationally acceptable guidance on sampling.
- 30. The Representative of ICUMSA highlighted the different aspects of sampling such as the taking of samples for which the IAM may be of assistance and the interpretation of results which is more an enforcement problem to be dealt with at the governmental level. In his opinion, CCMAS should point out the difficulties, i.e. whether the sampling procedures are obligatory. However, the CCMAS itself should not develop sampling procedures on its own.
- 31. As a result of the discussion it was agreed to wait for the outcome of the CCMAS Session. Participants felt that it would be too early for IAM to draw any conclusions at this stage.

# USSR PROPOSAL TO THE CCMAS FOR STANDARD METHODS OF ASHING (MINERALIZATION) OF FOOD PRODUCTS PRIOR TO THE DETERMINATION OF HEAVY METALS

- 32. The Meeting had before it the USSR proposal which had been submitted to IAM by the Codex Secretariat and the comments received from IDF and NMKL.
- 33. The Representative of AOAC informed the Meeting of the advantage of a general method of ashing which could be applied to a wide range of food products. The USSR proposal would shortly be submitted to AOAC with a view to its possible publication in the Journal of the AOAC.
- 34. The Representatives of IDF and NMKL informed the Meeting that their Organizations were working on general procedures for ashing which slightly differed from that specified in the USSR proposal. NMKL was organizing a collaborative test with its method, the result of which would be available in due curse.
- 35. Bearing in mind that according to its Terms of Reference, IAM itself does not select methods, the Meeting agreed to request the Codex Secretariat to identify Organizations which may be interested in the further study of the USSR proposal.

## REPORTS BY SPECIALIZED ORGANIZATIONS ON METHODS OF ANALYSIS AND SAMPLING REQUIRED BY THE CAC

36. Under this agenda item, Representatives of the various Organizations were invited to present statements on the relevant activities of the Organizations. Some of the Organizations had submitted written statements to the Secretariat, whereas others circulated their statement at the Meeting. AOAC had submitted contributions to most of the agenda items.

#### COCOA PRODUCTS AND CHOCOLATE

- 37. The Representative of OICCC tabled a comprehensive report on the activities of his Organization. He stressed the good cooperation that exists between OICCC and AOAC. Some of the methods developed by ISO/TC24/SC9 "Microbiology" and the ISO Standards on cocoa beans were of particular interest to the OICCC Analytical Committee and cited in the respective OICCC publications.
- 38. The Representative of OICCC noted that ISO 2292:1973 "Cocoa beans Sampling" would be revised and offered the assistance of his Organization for the revision work.

#### MILK AND MILK PRODUCTS

- 39. The Representative of IDF informed the Meeting of the Joint IDF/ISO/AOAC work on methods for the FAO/WHO Code of Principles for Milk and Milk Products and the work of IDF Commission E. He suggested that interested Organizations other than IDF, ISO or AOAC participate in this work and drew particular attention to the methods for the determination of iodine and tin.
- 40. The Representative of AOAC mentioned that his Organization had started work on a modified Mojonnier method for the determination of fat content, an item which was not a subject of the IDF/ISO/AOAC cooperation.

#### EDIBLE ICES

- 41. Referring to the IDF/ISO/AOAC work on edible ices, the Representative of IDF mentioned that four new methods had been agreed, i.e. total solids, colony count and two gravimetric fat determinations.
- 42. In response to a query by the Representative of the Codex Secretariat, the Representative of IDF informed the Meeting that the work on the review of the Codex Standard for Edible Ices could be initiated and that the Weibull-Berntrop method for the determination of fat content would be published shortly.

#### FATS AND OILS

- 43. The Representative of IUPAC mentioned the existing good cooperation between the Fats and Oils Commission of IUPAC and ISO/TC34/SC11 "animal and vegetable fats and oils" which had resulted in a large number of technically identical methods. In the same context, the good cooperation with AQAC was noted.
- 44. The Meeting also considered the question of multiple references in Codex Standards to identical methods developed by different Organizations. In accordance with current practice, technically identical methods developed by different Organizations can be referred to in Codex Standards in addition to the recommended method. However, it is the responsibility of the Organization concerned to state that its own method is technically identical.

#### FOOD FOR SPECIAL DIETARY USES

45. The Meeting considered the need for methods by the Codex Committee on Nutrition and Foods for Special Dietary Uses on the basis of a document prepared by a working group of this Codex Committee. It noted that various methods for calcium, sodium, potassium and magnesium were already available within AOAC and IDF. These methods had undergone collaborative tests in accordance with the provisions of the IUPAC Protocol and the future new edition of ISO 5725.

#### FRUIT JUICES

- 46. The Meeting regretted that IFJU had neither responded to the invitation to attend the IAM, nor submitted a written statement for this agenda item.
- 47. The Representative of AOAC drew attention to the AOAC methods for the determination of quinic, malic and citric acids in cranberry juice cocktail and applie juice which had been adopted.

#### PROCESSED FRUITS AND VEGETABLES

48. The Representative of the Codex Secretariat, referring to document CX/MAS 88/14, noted that suitable methods were still lacking for a number of products of interest to the Codex Committee on Processed Fruits and Vegetables.

#### PROCESSED MEAT AND POULTRY PRODUCTS

49. The Representative of ISO/TC34/SC6 "Meat and meat products" gave a brief account of the work of his Committee. Various participants expressed interest in methods for the determination of non-meat proteins, hydroxyproline, polyphosphates, as well as in some microbiological methods.

#### SUGARS

- 50. The Representative of ICUMSA informed the Meeting of recent developments within his Organization. At the ICUMSA meeting held in June 1986 a new working group was established to review the existing methods. The group will organize collaborative studies in order to validate the methods in accordance with the provisions of the IUPAC Protocol.
- 51. The Representative of IFG noted the cooperation between ICUMSA and IFG in the field of starch hydrolysis products which was also of interest to ISO/TC93 and AOAC.

#### STARCH HYDROLYSIS PRODUCTS

52. On behalf of the Secretariat of ISO/TC93, the Chairman informed the Meeting that this Committee had recently been re-activated. AOAC and IFG are actively participating in the work which is of particular interest to the Codex Committee on Sugars and the CEC.

#### CEREALS, CEREAL PRODUCTS, PULSES AND LEGUMES

- 53. The Representative of ICC presented a statement of the work of her Organization stressing the existing good cooperative with ISO/TC34/SC4 "Cereals and Pulses". She mentioned that in future all ICC methods would be validated in accordance with the provisions of the IUPAC Protocol. The Organizations participating in the IAM were also invited to attend the next ICC Symposium and Congress.
- 54. The Representative of ISO/TC34/SC4 introduced a document on the present work of this committee.
- 55. The Representative of AOAC informed the meeting of some matters of interest resulting from the recent Session of the Codex Committee on Cereals, Pulses and Legumes. The Committee was interested in methods for the determination of fat acidity of wheat flour, tannin content of sorghum, iron content by AAS, protein content by a near IR method etc.
- 56. The Representative of the Codex Secretariat informed the Meeting of the need for a suitable method for the determination of wheat flour in durum wheat flour. While an Italian and a French method for this purpose were known, the IAM might wish to consider the question of publication of such methods by an interested Organization.

#### **MICROBIOLOGY**

- 57. The Chairman introduced a report on the work of ISO/TC34/SC9 "Microbiology" and pointed out that this Committee develops general microbiological methods which could be adopted to the particular needs of individual commodities.
- 58. The Representatives of AOAC and NMKL mentioned some relevant activities of their Organizations. In this context, the need for a method for the detection of Brochothrix was mentioned. This contaminant may be present in vacuum packed meat products.
- 59. The Meeting also recognized the need for methods for the detection and enumeration of Listeria monocytogenes. An ISF/ISO/AOAC Group of Experts has already carried out trials with a method applicable for dairy products whereas NMKL and ISO were working on generally applicable methods.
- 60. The Representative of the Codex Secretariat emphasized the importance of collaboration between the various Organizations in the case of defining methods (Type I methods).

#### MINERAL WATERS

61. The Secretary mentioned that some of the chemical and microbiological methods standardized by ISO/TC147 "Water quality" presented an interest to the work of Codex on mineral waters. In this context, the Representative of the Codex Secretariat also underlined the need for methods for the detection and determination of radionuclides in natural mineral water.

#### CONTAMINANTS

- 62. The Representative of AOAC informed the Meeting of the establishment within IUPAC of a new working group on mycotoxins, elemental analysis by AAS and veterinary drugs (in particular for the facilitation of the transfer of tissue samples through customs).
- 63. The Representative of the Codex Secretariat drew attention to the various subsidiary bodies of CAC being interested in contaminants, i.e. the Codex Committees on Food Additives and Contaminants, Pesticide Residues, Veterinary Drugs and some of the Commodity Committees.

#### FOOD ADDITIVES

64. The Meeting had before it IDF document E-363:1988 which identifies priorities and criteria for the selection of methods.

65. The Representative of the Codex Secretariat confirmed that the methods required will have to be endorsed and validated by CCMAS. Given the large number of methods required, priorities will have to be set and a close collaboaration be established between the Organizations interested.

#### WINES AND SPIRITS

- 66. The Representative of OIV informed the Meeting that her Organization had started work on collaborative studies on the determinations of sugars by HPLC and organic acids by HPLC. A new Working Group had been established on the determination of ethyl carbamate. This group was studying a gas-chromatographic method with mass spectrometry detection and a HPLC method for extremely low contents (at the level of 16 ppb). A new edition of the OIV methods of analysis will shortly be issued.
- 67. The Representative of AOAC expressed interest in the determination of ethyl carbamate and offered the cooperation of his Organization on this subject.
- 68. The Representative of ICUMSA drew attention to the 1982 edition of the EEC methods of analysis for wines which took into account many of the existing OIV methods. In this context the NMR method for detection of chaptalization of wines was considered to be of particular interest.
- 69. The Representative of AOAC referred to a summary of collaborative studies carried out by OIV. This summary had been compiled by Dr. Junge of the Federal Health Office in the Federal Republic of Germany and published in the Journal of AOAC.

#### OTHER ACTIVITIES OF INTEREST TO THE INTER-AGENCY MEETING

- 70. The Representative of IDF introduced a document he had received from the Working Group on Methods of Analysis for Nutritional Labelling of the Codex Committee on Food Labelling which outlined the present situation with respect to methods of analysis and sampling in this field.
- 71. In view of the fact that methods of analysis for nutritional labelling may be of interest to various commodities, the Representative of IDF, supported by the Representative of AOAC, proposed that the agenda of the next IAM be arranged in a different manner. This would mean that items dealing with general subjects such as microbiology, contaminants and food additives will be grouped together and be separated from items that cover individual commodities on which the specialized Organizations will report.
- 72. The Meeting agreed to this proposal and requested the Secretary to amend the agenda of the next IAM accordingly.

#### STANDARDIZATION OF TERMINOLOGY IN THE FIELD OF METHODS OF ANALYSIS AND SAMPLING

73. The Chairman stressed the importance of uniform terminology for the work of CCMAS and mentioned the efforts of the different Organizations towards achieving agreement on a common language.

#### AOAC PROPOSALS FOR UNIFORM TERMINOLOGY USED IN COLLABORATIVE STUDIES

74. The Representative of AOAC mentioned that certain definitions had recently been amended following discussion that took place at the meetings of AOAC and IUPAC. An amended version of the terminology document would be circulated to all interested Organizations in due course.

#### COMPARATIVE DOCUMENT OF STATISTICAL DEFINITIONS

75. The Meeting had before it the comparative document of definitions as compiled by Mr. E. Nouat (ISO). Following circulation of this document to all Organizations no comments had been received.

76. The Chairman drew attention to ISO 3534:1977 "Statistics - Vocabulary and symbols" which is being revised by ISO/TC69. The new edition will be issued in separate parts and will take into account the comments and suggestions by other Organizations.

#### ANY OTHER BUSINESS

- 77. The Representative of IDF introduced IDF document E-372:1988 which outlined the present and future policy of his Organization with respect to proprietary laboratory techniques. The rapid development of laboratory techniques is likely to make the current standard methods obsolete and thus causes new problems to those involved in the standardization of traditional methodology.
- 78. The Representative of AOAC drew attention to an article on the use of test kits which had been published in "The Referee".
- 79. The Representative of the Codex Secretariat pointed out the problems that may arise in connection with the acceptance by governments of Codex standards containing Codex Type I or II methods based on the use of test kits. For example, test kits may not be readily available or may be withdrawn for commercial reasons.
- 80. The Meeting agreed that this matter should be discussed under a separate agenda item at the next IAM.
- 81. The Representative of the Codex Secretariat then referred to a document entitled "Limit of determination and limit of detection" which had been prepared by a Hungarian expert. He felt that this document should be discussed by members of the scientific community rather than the CCMAS.
- 82. The Representatives of AOAC and ICUMSA expressed interest in the document prepared by the Hungarian expert and suggested that it be submitted to the Royal Society of Chemistry of the United Kingdom and IUPAC, respectively.

#### DATE AND PLACE OF THE NEXT INTER-AGENCY MEETING

- 83. It was agreed that the next Inter-Agency Meeting should be held in Budapest, in November 1990, before the 1990 Session of the Codex Committee on Methods of Analysis and Sampling.
- 84. After thanking all participants, the Secretary, the interpreter, the staff of the Hungarian Office for Standardization and all who had contributed to the success of the Meeting, the Chairman closed the Seventh Inter-Agency Meeting.

## ATTENDANCE SHEET

# Seventh Inter-Agency Meeting Budapest (Hungary), 10 and 11 November 1988

ORGANIZATION	NAME	PROFESSION and ADDRESS
ISO	Mr G Castan	Délégué aux Programmes Priori- taires, AFNOR - Tour EUROPE 92080 PARIS LA DEFENSE
ISO Secretary of IAM	Mr K-G Lingner	ISO Central Secretariat, 1 rue de Varembé CH 1211, Gèneve 20
ISO	Mrs E Nagy	ISO/TC 34 Secretariat Magyar Szabványügyi Hivatal
ISO	Mr H W Schipper	Dutch Standards Institute (NNI) P.O.Box 5059 2600 GB Delft - Netherlands
ISO	Mr W G de Ruig	State Institute for Quality Control of Agricultural Prod. (RIKILT), P.O.Box 230 6700 AE Wageningen - Netherlands
ISO	Ms E Karsay	Secretary of ISO/TC 34/SC 4 Magyar Szabványügyi Hivatal
ICC	Ms H Reigner	Executive Secretary
IDF	Mr E Hopkin	Deputy Secretary General Int. Dairy Federation 41 Sqr Vergote, B-1040 Brussels
IFG	Mr D B Whitehouse	Quality Assurance Manager Cerestar SA/NV, Havenstraat 84, B 1800 Vilvoorde
NMKL	Ms H Wallin	Secretary General, Technical Research Centre of Finland, Food Research Laboratory, SF-02150 ESBO, Finland

## ATTENDANCE SHEET

# Seventh Inter-Agency Meeting Budapest (Hungary), 10 and 11 November 1988

ORGANIZATION	NAME	PROFESSION and ADDRESS
IOCCC	Mr H Vos	Populierenlaan, A NL-3235 LG Boschan Duin Analytical Committee: Dr E N Meursing
		Toren Laan 7 NL-1551 BK Westzaan
CC/MAS	Mr B Borszéki	Central Food Research Institute II. Budapest, Herman O. u. 15. H-1091
CC/MAS	Ms M Vámos	Central Food Research Institute II. Budapest, Herman O. u. 15. H-1091
EOQC	Mr P Molnár	Institute of Food Control 1095 Budapest, Mester u. 81
MSZH	Mr I Oláh	Head of Department, Hungarian Office for Standardization IX. Budapest, Üllői u. 25.
IUPAC	Ms M Jeránek	Research Institute for Veget. Oils and Detergents, Director 1106, Budapest, Maglódi u. 56
ICUNSA/AOAC	Mr R Wood	Ministry of Agriculture, Fisheries and Food, 65 Romney Street, London, SWIP 3RD, UK
AOAC	Mr J F Lawrence	Food Research Division, Health Protection Branch, Ottawa, Ontario, Canada KIA OL2
AOAC	Mr G W Diachenko	Chief, Food Formulation Branch Food and Drug Administration (HRF-413), Center for Food Safety and Applied Nutrition,
		200 C St. W.W. Washington, DC 20204

## ATTENDANCE SHEET

# Seventh Inter-Agency Meeting Budapest (Hungary), 10 and 11 November 1988

ORGANIZATION	NAME	PROFESSION and ADDRESS
AOAC	Ms G E S Cox	Chief Executive Officer Cox and Cox Investments 12006 Auth Lane Silver Spring, Maryland 20902
AOAC	Ms M Lauwaars	European Representative P.O.Box 153 6720 AD Bennekom The Netherlands
AOAC	Mr W Horwitz	Scientific Advisor HFF-7 Food and Drug Administration 200 C Street SW Washington, DC 20204, USA
FAO/WHO	Mr E Casadei	Food Standards Officer ESN / Codex Alimentarius via Terme di Caracalla 00100 Rome
FAO/WHO	Mr L G Ladomery	Food Standards Officer ESN / Codex Alimentarius via Terme di Caracella 00100 Rome
OIV	Ms B Mandrou	Professeur-Faculté de Pharmacie de Montpellier 34060 Montpellier Cedex France
Interpreter	Ms K Lomb	
	OBSERVERS	
	Mr. J.Daenen	Project leader Methods of Analysis for Food Control Services; Ministry of Health The Haque, Holland
	Mr.W.J.de Koe	Food Standards Officer Min.of Public Health Sir.Winston Churchillaan 362 2280 HK Ryswyk. The Netherlands