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

Bonn - Bad Godesberg, 25 - 28 January 1971

MR/B2725

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CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

Report of the Sixth Session

Bonn - Bad Godesberg, 25 - 28 January 1971

INTRODUCTION

1. The Codex Committee on Methods of Analysis and Sampling held its sixth session from 25 to 28 January in Bad Godesberg under the chairmanship of Prof. Dr. R. Franck. The chairman opened the session on behalf of Frau Käte Strobel, Bundesminister für Jugend, Familie und Gesundheit, who could not be present owing to other official engagements. The chairman welcomed delegations from 22 countries as well as observers from 10 international organizations. The list of participants, including officers from FAO, is contained in Appendix I.

ADOPTION OF AGENDA

2. The Committee agreed to adopt the Provisional Agenda (CX/MAS/70/A/2) with the following amendments: to discuss items 16 and 18 immediately following item 6 of the Provisional Agenda in view of the fact that these items represented matters of general importance in the Committee's work.

APPOINTMENT OF RAPPORTEURS

3. Mr. T.J. Coomes of the United Kingdom Delegation and Mr. Gosselé, the Belgian Delegate were appointed as rapporteurs.

MATTERS ARISING FROM THE REPORT OF THE SEVENTH SESSION OF THE CODEX ALIMENTARIUS COMMISSION

4. The representative of FAO advised the Committee of the system of uniform numbering adopted for all Codex documents by the seventh session of the Codex Alimentarius Commission (see Appendix III of ALINORM 70/43). It was noted that some time would elapse before the new document numbering system could be fully implemented as the Committee had before it documents prepared for previous sessions of this and other Committees.

5. The Committee was also informed that the Executive Committee would discuss the entire question of sampling at its sixteenth session in February 1971. The representative of FAO pointed out that this matter was under consideration and that every effort would be made to give sampling adequate attention, including the possible engagement of a consultant or the holding of a special session devoted to this topic. The Delegation of Poland pointed out that the whole problem of sampling had not yet received adequate attention by the Commission. The Delegation of the Federal Republic of Germany pointed out that the document on technical procedure of sampling as contained in Appendix VI, ALINORM 69/23, was still in force and should be discussed together with the respective ISO document (Standard layout and guide to the drafting of a method of sampling from a lot) at a later stage. The Committee agreed that the whole question of sampling required urgent and detailed study by the Executive Committee.

METHODS OF ANALYSIS FOR PRESERVATIVES IN FOOD

6. The Committee had before it a document prepared by the Netherlands Delegation on the methods of analysis for antioxidants and preservatives not permitted in food (CX/MAS/70/C/3) 1/ together with a number of documents containing proposed methods of analysis for preservatives in fruit juices. As regards the latter, the Committee agreed with the proposal by the chairman to discuss item 5(a) of the agenda (Preservatives in Fruit Juices) together with item 10, which dealt with methods of analysis in the Standards for fruit juices. In view of the fact that the document prepared by the

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1/ See also previous document prepared by the Netherlands SP 10/101 CODEX/ANALYS/67/10 and comments in MA/68/11 Rev. September 1969 and CODEX/ANALYS/69/B/6.

Netherlands was received only at the beginning of the present session and that delegations would not have had the opportunity to study its contents, the Committee agreed not to discuss the proposals contained in the paper in detail. Some delegations pointed out that, in the case of non-permitted additives only qualitative methods were necessary. However, permitted additives would require quantitative methods of estimations.

7. Several delegations drew the Committee's attention to the availability of such quantitative methods. The Delegation of the U.S.A. undertook to make available to the Secretariat references to the latest official AOAC 1/ methods for incorporation into the paper. The Delegation of the Netherlands agreed to revise the document and re-submit the revised version in this respect to the next session of this Committee. Participants were requested to send to the Delegation of the Netherlands any methods which they had found to be appropriate.

METHODS FOR THE DETECTION AND IDENTIFICATION OF COLOURS IN FOOD

8. The Committee had before it a paper prepared by the Delegation of the United Kingdom (CX/MAS/70/C/4). In introducing the paper the Delegation of the United Kingdom pointed out that the methods contained in the paper had so far been verified only in model systems and that the problem of any degradation of the colours in food needed further investigation. Furthermore, standard methods of extraction would also need elaboration. Both the extraction procedure and the methods of separation and identification would need adequate collaborative study before a suitable method could be finalized. It was pointed out that Table 1 of the document listing the food colours permitted in various countries required revision; participants were requested to notify the Secretariat of the exact legal status of all colours in their countries at the present time.

9. A number of delegations were of the opinion that the methods described in the United Kingdom paper required collaborative study and expressed their willingness to participate in such an undertaking. The Delegation of the United Kingdom agreed to organize such a collaborative study and to provide standard samples for those interested. The Delegation of the U.S.A. was of the opinion that before participating in such a programme of collaborative work, countries should try out the United Kingdom methods on colours of importance in their countries in comparison with their own methods. Participants trying the method were requested to submit any comments arising from their trials to the United Kingdom by July 1971. The Committee noted that the Codex Alimentarius Commission had adopted a list of colours for use in food (see Appendix VII to ALINORM 70/43) and also noted that the Secretariat had provided a list of colours which had been shown to be unsafe for food use by the Joint FAO/WHO Expert Committee on Food Additives (see CX/MAS/70/A/5).

METHODS OF ANALYSIS OF GENERAL APPLICATION TO THE CODEX ALIMENTARIUS

10. The Committee discussed the paper (MA 68/1) prepared by the Delegation of Poland, dealing with the "Table of Contents of the General Part of the Methods of Analysis" to be included in the Codex Alimentarius as well as a paper (CX/MAS/70/C/6) containing comments on this document received from governments.

11. There was a general discussion on the establishment of general methods of analysis as a fundamental part of the work of the Committee. Some delegations were of the opinion that the document prepared by Poland should be regarded as a check-list for future work, on the basis of which priorities could be established. The Committee requested the Delegations of Poland to retain responsibility for this work and to prepare, in collaboration with the Delegation of the Federal Republic of Germany, a paper for the next session of the Committee setting out the specific analytical sections in order of priority. The "Table of Contents" refered to in paragraph 10 above is given in Appendix II.

1/ General: JAOAC, 48 (1965), 498-92; AOAC (1970) 20.006-8
Salicylic acid: AOAC (1965), 27.073, 27.075-6; AOAC (1970) 20.084
Thiourea: AOAC (1965), 27.087-8
Boric acid: AOAC (1965), 27.010, 6.073, 27.014; AOAC (1970), 20.034-8, 20.029, 3.079-3.080, 20.033

GENERAL METHODS FOR THE DETERMINATION OF METALLIC CONTAMINANTS

12. The Committee had before it a paper prepared by Canada (CX/MAS/70/C/2) on the determination of certain metallic contaminants in food. The Delegation of the Federal Republic of Germany stressed the need to establish internationally acceptable methods of determination of mercury and certain other metals. The Committee agreed that, for the present time, it was sufficient to consider the determination of total mercury content. The Committee considered that methods for the determination of organic mercury compounds were not yet standardized, although it was recognized that methyl-mercury compounds were of particular interest from the toxicological point of view.

13. As regards the determination of metallic contaminants a number of delegations expressed preference for the atomic absorption spectroscopic method which, in their opinion, had yielded satisfactory results. It was generally agreed that the main problem in the determination of metallic contaminants was the method of digestion of the sample. The Delegation of Canada was requested to include in their paper references to results of collaborative work. The Committee expressed its appreciation to the Delegation of Canada and requested participants at the session to send their detailed comments on the methods of analysis contained in the paper to Mr. F. Sheffrin, Chairman, Interdepartmental FAO Committee, Department of Agriculture, Ottawa 4, Ontario Canada, by July 1971. On the basis of comments received the Committee would be in a position to reach conclusion at its next session.

UNIFORMITY OF METHODS OF SENSORY ANALYSIS

14. The Committee examined a paper prepared by ISO (CX/MAS/70/C/7) together with the original paper MA 68/2 prepared by Poland. The representative of ISO stressed the need to bring uniformity into the organoleptic examination of the various quality criteria. The Delegation of Poland drew the Committee's attention to the progress which had been made in this field during the last few years.

15. The Delegation of the Federal Republic of Germany referred to a paper containing a bibliography of methods of sensory analysis published by the Swedish Institute for Food Preservation Research. The Committee requested the Delegation of Poland to continue acting as rapporteur in this field and requested the Secretariat to take steps to obtain the Swedish document and to make it available to this Committee for future reference. The Committee recalled its previous discussion concerning collaboration with ISO in this field (paras 28 - 29, ALINORM 70/23).

METHODS OF ANALYSIS IN STANDARDS FOR FATS AND OILS

Determination of the water content of margarine (Standard at Step 9)

16. The Committee received a report from the Delegation of the Netherlands (CX/MAS/70/C/1) on a collaborative study involving the determination of water content in salted and unsalted margarine using the method which appeared as Appendix IX A - Draft Standard B-9 (1968) to Report of the 11th Session of the Joint FAO/WHO Committee of Government Experts on the Code of Principle concerning Milk and Milk Products (pages 124-125) and a method elaborated in the Netherlands involving the addition of sand. On the basis of the collaborative study the Delegation of the Netherlands recommended the method given in Appendix I to the above paper.

17. The Committee discussed desirability of cooling the dried sample in a dessicator before weighing. The Delegation of the Netherlands pointed out that the errors introduced by cooling the dried sample in air, were insignificant whereas the use of dessicators for this process led to difficulties in attaining a constant weight due to readsorption of water from the equilibriated air above the dessicant. This was supported by a number of delegations. It was understood, that the weighing would normally be carried out under average atmospheric conditions in the laboratory. Extremes of humidity and temperature would need careful control. The Committee suggested coordinating with the IDF, ISO, AOAC group working on methods for the determination of water in dairy products, including butter. The Committee agreed that the method elaborated in the Netherlands (CX/MAS/70/C/1) be referred to the Codex Committee on Fats and Oils for consideration.

Determination of contaminants in margarine (Standard at Step 9)

18. The Committee considered the proposal by the Secretariats of FAO and of the Codex Committee on Fats and Oils that the methods endorsed for the determination of iron, copper, lead and arsenic in fats and oils should also be included in the standard for margarine. The Committee was in agreement with the proposal and decided to endorse the following methods for margarine:

Iron: CAC/RM 14-1969 Determination of Iron Content (FAO/WHO Codex Alimentarius Methods of Analysis for Edible Fats and Oils, p. 17).

Copper: AOAC, 1965, 24.023 - 24.028 (Carbamate method)

Lead: AOAC, 1965, 24.053 (and 24.008, 24.009, 24.043j, 24.046, 24.047 and 24.048) (Dithizone procedure)

Arsenic: AOAC, 1965, 24.011 - 24.014, 24.016 - 24.017, 24.006 - 24.008 (Silver diethyldithiocarbamate method)

Determination of tocopherols in olive oils (Standard at Step 9)

19. The Committee endorsed the method for the determination of tocopherols as described in the Recommended International Standard for Margarine (Method CAC/RM 18-1969, page 23 of CAC/RS 32-1969) for olive oils as proposed by the Codex Alimentarius Commission (see paragraph 77 of ALINORM 70/43). The Delegation of the U.S.A. drew the Committee's attention to the fact that a new method to be published in the March 1971 issue of the Journal of the AOAC had been subjected to collaborative studies with favourable results involving a wide variety of foods including foods for special dietary uses.

METHODS OF ANALYSIS IN STANDARDS FOR EDIBLE FUNGI AND FUNGUS PRODUCTS, DRIED EDIBLE FUNGI AND FRESH FUNGUS "CHANTERELLE" (at Step 9)

20. The Committee had before it a synopsis of methods of analysis for edible fungi and fungus products prepared by Poland (CX/MAS/70/C/5) and a paper containing government comments on the synopsis (CX/MAS/70/B/2). The Committee noted that the methods of analysis for the recommended Standards for fungi now at Step 9, had been circulated to governments for comment as recommended by the Commission (para 90(d), ALINORM 70/43). The methods endorsed by the Codex Committee on Methods of Analysis and Sampling would require to be considered by the Coordinating Committee for Europe and the Commission before publication as recommended international methods.

Determination of mineral impurities

21. The Committee noted that the various standards for fungi defined mineral impurities as residues insoluble in hydrochloric acid after ashing. It was therefore considered that the ISO method (R 763) was strictly relevant to the above provision while this was not the case with the AOAC method. The Delegation of the U.S.A., referring to the discussions on mineral impurities at the fifth session of this Committee (see ALINORM 70/23 paras 52, 53, 58, 59, 63 and 67) was of the opinion that, while the ISO method may well have greater relevance to the provision in the standards as drafted, it was possible that the original intention of the Coordinating Committee was to provide for a measure of the extraneous soil and sand associated with the products, for which the AOAC method based on flotation might be more appropriate. The Committee endorsed the ISO method R 763, with the amendments proposed at the fifth session of the Committee (CL 1970/5 para B), provided the Commodity Committee was satisfied that the method was appropriate to the provision concerned.

Determination of salt content

22. The Delegation of the U.S.A. drew the Committee's attention to a general potentiometric method for the determination of sodium chloride which will be published in the Journal of the AOAC (March 1971) and bearing the reference numbers 32.A01 - 32.A05, copies of which have been sent to the Secretariat. The Committee agreed that this method be recommended for consideration by the Coordinating Committee for Europe and did not endorse the method AOAC 1965, 31.009.

Determination of water, acetic acid, lactic acid, citric acid and sugars content

23. The Delegation of the Netherlands was of the opinion that more specific methods (e.g. the use of enzymes) were required to distinguish between the various organic acids. The Committee noted that, with the exception of the standard for sterilized fungi, no requirement to distinguish between these acids arose since only one acid was provided for in each individual standard. The Committee was of the opinion that the methods of ISO (R 750), IFJU (No. 3/1968) and AOAC (1965, 20.042) were adequate for the purpose of the various standards for fungi.

24. The methods recommended in CX/MAS/70/C/5 were endorsed (see Appendix III).

METHODS OF ANALYSIS IN STANDARDS FOR FOODS FOR SPECIAL DIETARY USES

Methods of analysis in standard for dietary foods with low sodium content at Step 8

25. The Committee considered proposals made by the sixth session of the Codex Committee on Foods for Special Dietary Uses (ALINORM 71/26, Appendix III, para 5).

Determination of sodium content

26. The Committee had before it CCDF/69/6 (with an addendum) and CX/MAS/70/B/1 (with 2 addenda) detailing proposals for methods of determination of sodium in low-sodium foods as well as current comments on this subject. As regards wet-ashing versus dry-ashing procedure, the Delegation of the Netherlands pointed out that, in many cases, (e.g. milk and milk products), no ashing at all would be required. It also mentioned that dry-ashing would result in loss of sodium chloride by volatisation. The Delegation of Hungary informed the Committee that in the Lindner and Dworschak method, the time required for the digestion of a 10 g sample would be up to 7 hours, rather than the 3 - 4 days described in the method. The Delegation of the Federal Republic of Germany was in favour of dry-ashing. The Delegation of the U.S.A. described a collaborative study they had undertaken comparing both methods of ashing. Each method had given an identical result. The Delegation of Switzerland pointed out that the flask used for this method should be of quartz, when wet-ashing was employed.

27. The Committee considered methods involving wet- and dry-ashing but did not come to any conclusion. It was agreed that the methods in question be reexamined at the seventh session in the light of comments received.

Determination of potassium, calcium, magnesium, ammonium, phosphorus, silica and choline

28. The Committee examined the proposals made by the Codex Committee on Foods for Special Dietary Uses regarding the determination of potassium in foods other than salt substitutes and the determination of potassium, calcium, magnesium, ammonium, phosphorus, silica and choline in salt substitutes as such. The Delegation of the U.S.A. pointed out that there was no satisfactory method for the determination of choline. On the proposal of the Delegation of Canada it was agreed that other methods should also be proposed for the detection of such substances which were either not permitted, but nevertheless used as salt substitutes, or substances which were not included in the standard. Methods for the detection of glutamic acid were also

regarded as essential although presently no restriction on the use nor a limit for its total amount had been laid down in the Standard. The Delegation of the U.S.A. advocated the AOAC method 1970, 20.149 - 20.151 (determination of glutamate). The Committee decided to postpone any decision regarding the methods proposed for determination of these compounds to the seventh session, pending further proposals by the Commodity Committee.

METHODS OF ANALYSIS IN STANDARDS FOR FRUIT JUICES

29. The Committee had before it the Report of the seventh session of the Joint ECE/Codex Alimentarius Group of Experts on Standardization for Fruit Juices (ALINORM 71/14, paras 68 and 69). The Committee noted that important provisions of the standards were not yet covered by appropriate methods of analysis. In particular, the following would seem to be required:

(i) minimum content of fruit ingredient (in apricot, peach and pear nectars);

(ii) soluble fruit solids of fruit juices (exclusive of added sugars), where the addition of sugars was permitted by the standards;

(iii) degree of concentration of fruit juices;

(iv) differentiation between juices obtained by direct expression and those prepared by reconstitution using concentrates.

The Delegation of Portugal was of the opinion that the French version of the Standards should refer to "Résidu sec" instead of "Solides solubles".

30. The Committee took note of the work already undertaken in Israel involving different approaches to these problems (determination of amino-acids, vitamins of the B group, pigments, anti-serums) as recorded in the above Report (ALINORM 71/14). The Delegation of Spain mentioned several publications available in Spain concerning the determination of mineral content (Na, K, Mg etc..), protein and amino-acid contents in citrus juices as well as in soft drinks containing citrus juices. The Committee was informed that these methods would permit differentiation between fruit juices from preparations containing other substances with a good degree of precision.

31. In connection with methods needed to determine purity of fruit juices, the Delegation of Canada informed the Committee that it had already applied analytical methods for the estimation of potassium, polyphenolics and free amino acids to reconstituted fruit juices and had used the results obtained by these methods as evidence proving excessive dilution in a court case.

32. The representative of AOAC also mentioned five new AOAC methods dealing with the estimation by GLC techniques of several components in fruit juices.(Free amino acids, sugars, organic acids, carbohydrates, flavanones. See Abstracts, 84th Annual Meeting, October 12-15, 1970, AOAC, p.12). The chairman of the Commission on Methods of Analysis of the International Federation of Fruit Juices Producers pointed out that such research was difficult, when applied to reconstituted fruit juices. The Committee agreed that the papers of the Canadian and the Spanish Delegations as well as the five AOAC methods should be sent to the Secretariat for transmission to, and examination by, the Group of Experts.

Expression of figures per kilogramme

33. The Committee noted that figures in the standards were expressed in terms of mass. On the other hand, most of the methods of analysis measured the sample for examination in terms of volume. The Group of Experts had stated that (ALINORM 71/14, para 70) despite the views of the Commission (ref. ALINORM 70/43 para 108(2)) they did not wish to change the standards at this stage.

34. The chairman of the Commission on ... Methods of Analysis of the International Federation of Fruit Juices Producers proposed that the results of analysis could be calculated and expressed per kg.by using the relative density of the product

(Method IFJU 1, rev. 1968, already endorsed ALINORM 70/23, para 41). The Committee agreed. (See Appendix IV). The Committee also agreed to an alternative proposal that in any method which specified a volume of fruit juice to be taken for the determination, a given weight could be used instead. (See Remark in Appendix IV). The representative of the IOV was strongly opposed to the decision regarding relative density. She indicated that, in this case, density (symbol ρ, 'masse volumique' in French) should be used.

Test for fermentability and methods of analysis for preservatives in fruit juices

35. The Committee was informed that the IFJU method No. 18, fermentation test was not suitable for citrus juices because the ethereal oils present inhibit fermentation. It was noted however that this method would be valid for non-citrus juices while a modified IFJU method was being developed. The Committee agreed that, when complete, this method should be sent to the Secretariat for examination at the seventh session of the Committee.

36. The Committee's attention was drawn to the ISO method for the determination of free, combined and total sulphur dioxide. The Committee agreed that any newly developed method for determination of the total SO_2 as well as the results of collaborative studies on this question should be sent to the Secretariat.

37. As regards other preservatives in fruit juices the Committee agreed that they would be dealt with by the general methods for preservatives in food.

General methods for water capacity and fill of containers in all Standards for fruit juices at Step 8

38. The Committee endorsed the method published in the Almanac of the Canning, Freezing, Preserving Industries, 55th Edition 1970 (p. 131-132), E.E. Judge and Sons, Westminster MD (U.S.A.). The method appears as Appendix V to this Report.

Determination of titratable acid (total acid) for citrus fruit juices at Step 8

39. The Committee endorsed the IFJU Method No. 3 (1962) on the understanding that results were expressed as anhydrous citric acid in g/kg. The representative of OIV pointed out that results might be better expressed in terms of milliequivalents per kilogramme.

METHODS OF ANALYSIS IN STANDARDS FOR PROCESSED FRUITS AND VEGETABLES

40. The Committee considered the document CX/MAS/70/A/3 which gave the proposed methods of analysis in standards for processed fruits and vegetables. At the suggestion of the Delegation of Poland the Committee agreed to request the Commodity Committee to prepare a synopsis of all the available analytical methods for processed fruits and vegetables so that suitable methods for the determination of criteria in the standards can be selected.

Mineral impurities (in Canned Strawberries at Step 8)

41. During discussions the Delegation of the U.S.A. informed the Committee that collaborative studies had been carried out recently by the AOAC and it was found that the determination of mineral impurities as given in the ISO Recommendation R 762 was not satisfactory. As a result of this collaborative study a new method had been developed and specifically applied to the analysis of mineral impurities in strawberries and spinach. This had been submitted to the Secretariat. The Delegation of the Federal Republic of Germany supported the views of the U.S.A. The Committee did not therefore endorse the Method ISO R 762 and decided to send its comments, together with all the data provided by the U.S.A. Delegation, to the Commodity Committee for reconsideration.

Calcium (in Canned Strawberries at Step 8)

42. The Committee did not reach any conclusions concerning the oxalate precipitation method AOAC (1965) 20.028 and referred the matter to the Commodity Committee for new proposals to be given in the synopsis (see para 40 of this Report).

Calcium (in Canned Green Peas at Step 6)

43. The Committee endorsed the EDTA method CAC/RM 38-1970 "Determination of calcium in canned vegetables" published in "FAO/WHO Codex Alimentarius methods of analysis for processed fruits and vegetables". The Committee was informed by the Delegation of the U.S.A. that this method has now been introduced in the 1970 edition of the AOAC (ref. 32014 - 32016).

Determination of water capacity of containers (General method for all canned fruits and vegetables at Steps 9 and 8)

44. The Committee had before it the method for determination of water capacity of containers proposed by the Commodity Committee (reference ALINORM 71/20, e.g. Appendix II para 7.6). The Committee decided that this method could be used as a general method and endorsed it for all canned fruits and vegetables at Steps 9 and 8). In this connection the Delegations of the Netherlands and the Federal Republic of Germany also suggested that the Commodity Committee might investigate the use of the method given in ISO R 90.

Determination of Proper Fill in lieu of Drained Weight (for Canned Peas at Step 6)

45. The Committee considered the method recommended by the Commodity Committee and reproduced in ALINORM 71/20, Appendix V, Annex 1. There was some discussion regarding the need for a specific method such as this for canned peas, rather than a General method such as the "Determination of Drained Weight" already adopted for other fruits and vegetables. The Delegation of U.S.A. pointed out that this was a special method for canned peas only and was particularly useful because of its comparative simplicity when compared with the method for the determination of Drained Weight and that the method had been in use for a considerable time. The Delegation of Canada proposed that in view of the fact that the method was applicable only to canned peas, it could be re-worded by replacing the word 'content' by the word 'peas'. The Committee decided to endorse the method as originally recommended, on the understanding that it might at some stage be possible to use it as a general method for some other canned fruit and vegetables. The Delegation of the Federal Republic of Germany wished to reserve their position until the text of the method was available in its revised form.

Determination of drained weight for Canned Mushrooms in Oil at Step 8

46. The Commodity Committee had recommended to replace the method for determination of washed drained weight endorsed by the Committee at its fourth session. This was necessary as canned mushrooms packed in oil could not be washed with water effectively as recommended in the procedure for determining washed drained weight. The Commodity Committee had therefore revised the standard to provide for a limit on drained weight rather on washed drained weight. The Committee decided to endorse the method CAC/RM 36-1970 in FAO/WHO Codex Alimentarius Method of Analysis for Processed Fruits and Vegetables for determination of drained weight.

Determination of washed drained weight for Canned Mushrooms (in "sauce packs" at Step 8)

47. The Commission at its seventh session had requested the Committee to reconsider the method for the determination of washed drained weight for canned mushrooms in "sauce packs" (ALINORM 70/43 para 90(d)). The Committee on reconsideration did not visualize any difficulty in carrying out this determination using water as the washing agent and decided to maintain its earlier endorsement of the method given in ALINORM 69/23, Appendix IV page 3.

Determination of Natural Soluble Tomato Solids (for Processed Tomato Concentrates at Step 6)

48. The Committee had before it document MAS/70/B/3. At its fifth session the Committee had endorsed the method proposed by the Commodity Committee (JAOAC 50, 1967 p. 690) but referred back the title of the method to the Commodity Committee for clarification as there was some confusion between the terms "natural soluble solids" given in the standard and "total tomato solids" determined by the method (see ALINORM 70/23 para 56). The Codex Committee on Processed Fruits and Vegetables at its seventh session had discussed this title and confirmed that the requirements of the standard were "natural soluble tomato solids" and not "total tomato solids".

49. The Delegation of U.S.A. expressed the opinion that the proposed method was somewhat empirical in nature, and that the relevant provision in the standard was based on the method of analysis proposed by the Commodity Committee. If therefore, any change was proposed in the method of analysis, this would involve a necessary change in the standard as well. The Committee was further informed that this method had now been incorporated in the latest edition of the AOAC. The Committee confirmed its previous endorsement of the method proposed by the Commodity Committee as given in AOAC, 11th edition, 32.008-32.010.

Determination of salt (for Processed Tomato Concentrates at Step 6)

50. At its fifth session the Committee had endorsed a potentiometric method (AOAC, 1965, 6.103-6.105)but on the understanding that more details were needed (ALINORM 70/23, para 57). The Committee was informed that during the past year the AOAC had conducted a collaborative study of the potentiometric method for the determination of sodium chloride in canned vegetable products including processed tomato paste. During this study additional details were given including the preparation of the sample as indicated by the Committee at its fifth session (ALINORM 70/23 para 57). Besides tomato concentrate the study included analysis on samples of peas sweetened with sugar, green beans, yellow corn, sauerkraut, dill pickles, and olives with pimentos. As a result of this study a detailed method would now be published under reference JAOAC March 1971, 32.A01-32.A05. The Committee decided to endorse the revised method.

Moisture determination for Raisins at Step 6

51. The Committee considered document CX/MAS/70/B/3. At its fifth session, the Committee had pointed out that the proposed AOAC method needed to be further elaborated especially with regard to sample preparation and the type of drying oven to be used (ALINORM 70/23 para 62). On the basis of the above remarks the AOAC had reviewed the experimental work on the method and confirmed that the directions given in the method were sufficiently detailed to provided reproducible results provided that the directions were followed exactly. The Committee confirmed its endorsement of the method as reproduced in the latest edition of the AOAC methods 1970,22.012 para 2.

52. The Delegation of Poland briefly informed the Committee concerning a synopsis of the methods elaborated by ISO/TC 34/SC 3 Fruits and Vegetables. At the suggestion of the U.S.A. Delegation it was decided that the Secretariat should request the Polish Secretariat of the ISO sub-Committee to provide also the results of any collaborative studies, wherever these had been carried out on individual methods. This information should be compiled and placed before the next meeting of the Committee for information.

METHODS OF ANALYSIS IN STANDARDS FOR QUICK FROZEN FOODS

Determination of mineral impurities, such as sand (in quick frozen Strawberries at Step 8, quick frozen Peaches at Step 5, quick frozen Bilberries at Step 5)

53. The Committee made the same reservations concerning ISO Recommendation R 762, as it had already made for the determination of mineral impurities in Canned Strawberries at Step 8 (see para 41 of this Report) and requested the Joint ECE/Codex Alimentarius Group of Experts on Quick Frozen Foods to obtain the new method elaborated by the U.S.A. Delegation and consider whether they might not propose this in place of R 762.

The U.S.A. Delegation mentioned that this method had already been successfully used for quick-frozen spinach.

Determination of total soluble solids content (in quick frozen Strawberries at Step 8, quick frozen Peaches at Step 5, quick-frozen Bilberries at Step 5)

54. The Committee endorsed the method proposed by the Group of Experts (see Appendix VI to this Report).

Determination of alcohol-insoluble solids content (in quick frozen Peas at Step 9)

55. The Committee was informed by the Delegation of the U.S.A. of the results obtained during a collaborative study undertaken on the method for quick-frozen peas endorsed at its fifth session (see para 51 and Appendix IV page 7 to ALINORM 70/23) at the levels prescribed in the standard (19-23%). This method had been found as valuable both for peas of good quality and for peas of poorer quality. The Committee confirmed its previous endorsement of this method.

METHODS OF ANALYSIS IN THE RECOMMENDED EUROPEAN REGIONAL STANDARD FOR HONEY at Step 9

Diastase activity in honey

56. The Committee reconsidered the possible revision of the method for diastase activity in honey (CAC/RS 12-1969, para 6.7) according to a proposal made by the Delegation of the Netherlands at its fifth session (see ALINORM 70/23 para 83). The Committee had before it the BENELUX method (CODEX/ANALYS/69/C/4, February 1970). Some delegations were in favour of changing the method in the honey standard but, after discussion,the Committee agreed that no basic modifications would be made to this method which had already been sent to governments for acceptance, since such a modification would necessitate the amendment of the provision for diastase activity in the standard for honey.

57. The Committee, nevertheless,recognized unanimously that the method in the standard should be amended as regards the preparation of soluble starch and the determination of the moisture content of this soluble starch. For this purpose the Committee agreed as to the paragraphs 1.1 and 1.2 of the BENELUX method. The Committee also recognized that the maintenance of hydrated starch at constant quality during storage was of importance and requested the Delegation of the Netherlands to specify the percentage moisture content as well as storage requirements for the soluble starch in any revised version of its text. The Committee agreed that this new text should be sent by the Secretariat to the Coordinating Committee for Europe, for examination at its eighth session prior to submission to the Commission as a proposed amendment.

METHODS OF ANALYSIS AND SAMPLING IN STANDARDS FOR FISH AND FISHERY PRODUCTS

Determination of net contents of products covered by glaze (for frozen Fillets of Cod and Haddock at Step 8, frozen Fillets of Ocean Perch at Step 8, and frozen Fillets of Plaice and similar flat fish at Step 6)

58. The Committee considered the method proposed by the Commodity Committee in Appendices to ALINORM 71/18. Several delegations were of the opinion that the deglazing of the product should be conducted in such a way that no re-glazing could occur by the wash-water film during draining. For this reason some delegations emphasized the need for the use of a towel or a paper towel to remove all external water from fish fillets. The Committee agreed to endorse the proposed method and to ask the Codex Committee on Fish and Fishery Products whether they had considered the use of a towel for this purpose.

59. On a proposal made by the Delegation of Poland the Committee agreed to ask both the Codex Committee on Fish and Fishery Products and the Joint ECE/Codex Alimentarius Group of Experts for Quick Frozen Foods to prepare a synopsis of all methods of analysis used in the standard they had been developing for frozen products.

METHODS OF ANALYSIS AND SAMPLING IN STANDARDS FOR COCOA PRODUCTS AND CHOCOLATE at Step 4

60. The Committee considered the document CX/MAS/70/A/4 containing the methods of analysis and sampling proposed by the Commodity Committee. The Committee was informed by the chairman of the OICC Expert Committee about the collaboration between the OICC, AOAC, and IUPAC in the development of standard methods of analysis in the field of cocoa products and chocolate. The Committee was also informed that AOAC was collaborating further with OICC in other methods of analysis to be included in the standards. The important work undertaken by ISO in its field was also emphasized.

Sampling

61. On the suggestion of the Delegation of Poland and in view of the fact that the ISO method of sampling cocoa beans - ISO/TC/34/416/513 was still in the process of elaboration the Committee agreed to postpone any decision on endorsement of methods of sampling.

Methods of Analysis

62. The Committee decided to endorse the following methods of analysis proposed by the Commodity Committee for the time being only, thus permitting interested laboratories to study these methods of analysis in greater detail. The Committee could then reconsider the methods at its seventh session.

(a) Preparation of the sample (cocoa butter) - IUPAC II.A.1

(b) Refractive index (cocoa butter) - IUPAC II.B.2
The Committee recommended that the value at 40°C should be given in the standard.

(c) Free fatty acids (cocoa butter) - IUPAC II.D.1

(d) Saponification value (cocoa butter) - IUPAC II.D.2

(e) Iodine value (Wijs) (cocoa butter) - IUPAC II.D.7.3

(f) Unsaponifiable matter (cocoa butter) - IUPAC II.D.5.2 (light petroleum)

(g) Iron (cocoa butter) - BS method: CAC/RM 14-1969

(h) Copper (all cocoa products) - AOAC (1965) 24.023

(i) Arsenic (all cocoa products) - AOAC (1965) 24.011 (24.015, 24.017)

(j) Lead (all cocoa products) - AOAC (1965) 24.053

The Committee decided that at present, methods (g) to (j) could be adopted. At a later date, consideration could be given to the methods based on atomic absorption spectrophotometry (in relation to the decision on item 16 b. See para 13 of this Report).

(k) Moisture content - (cocoa powder, cocoa,fat-reduced cocoa powder, fat-reduced cocoa)

The Committee agreed to the method AOAC (1965) 12.001 - 12.002 which did not use sand. It was however felt that in case of samples of certain cocoa products of high fat content, the method OICC 3-E/1952 could be more

useful as it used sand. Sand would help in preventing the formation of a fat-layer which may interfere with drying.

(l) Moisture content (loss on drying) (cocoa beans) - ISO 414/515 (to be finalized)

(m) Total fat - Milk chocolate, milk couverture chocolate, skimmed milk chocolate, cream chocolate, skimmed milk couverture chocolate, milk chocolate vermicelli, milk chocolate flakes — Text proposed by OICC (AOAC) and published as AOAC (1970) 13.035-13.036

OTHER BUSINESS AND FUTURE WORK

Solvent residue analysis

63. The Delegation of the Netherlands enquired concerning the methods of determination of solvent residues in food. The Secretariat informed the Committee that the main reason for not placing these methods before the Committee for endorsement was to avoid overloading the Agenda. The representative of FAO pointed out that the Joint FAO/WHO Expert Committee on Food Additives had considered solvent residue and had recommended methods for analysis for these residues in food. It was understood that eventually these methods would be placed before this Committee for endorsement.

Work priorities

64. The Secretariat pointed out that, in view of present work load, it was desirable to establish priorities to facilitate drawing up the agenda for the next session. It was agreed that the following items should receive first priority:

(a) General methods of analysis for preservatives, antioxidants, colours, etc..;

(b) Methods of analysis for foods for special dietary uses (e.g. foods for infants and children);

(c) Methods of analysis in Codex Standards at advanced Steps of Procedure (e.g. meat and meat products, mineral waters);

(d) Methods of analysis for solvent residues;

(e) Taking of samples.

Circulation of Codex documents

65. The Delegation of the U.S.A. raised the question of certain synopsis and other methods of analysis in extenso, which he had sent to the Secretariat for circulation to the Committee. He understood that these had not been circulated owing to the fact that the large cost of this operation could not be borne by the 1970/1971 allocation of printing within FAO or the facilities of the Secretariat of the Committee. He asked, therefore, whether the Committee could give him permission to circulate these documents (appropriately numbered) to the heads of delegations. The Committee agreed

that this could be done and the chairman suggested that all delegates could circulate such methods of analysis as were appropriate to the Committee and which were in their possession to other delegations.

66. The Delegation of the United Kingdom thought that this procedure would raise a question of principle and would need an enormous coordinating effort on the part of the Secretariat. They thought it appropriate that if the facilities were such that the circulation of such papers by the normal Codex procedure was in jeopardy, the attention of the Commission should be drawn to this fact and their guidance requested.

Pollen analysis of honey

67. The Delegation of Switzerland informed the Committee that an atlas for the identification of pollen types had been published by J. Louveaux (Services de Répression des Fraudes, Publisher, 42 bis, Rue le Bourgogne, 75 Paris VII - France) and referred also to a reprint from the Bee Research Association M58, Methods of Melissopalynology, Inter. Commission for Bee Botany of the I.U.B.S.

Sources of error

68. The Committee was of the opinion that it would be desirable to examine at a later date the variations of different analytical methods since in some cases the sampling errors are much greater than the analytical errors.

TIME AND PLACE OF THE NEXT SESSION

69. The Committee agreed that the next session should take place approximately two months after the last Commodity Committee, which would be submitting methods to be endorsed, had met. The Committee was informed by the Secretariat that the Commission had decided to meet at intervals of more than one year. It was possible that other Codex Committees might adopt this timescale.

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EXAMPLES

To explain the practical use of the general part of "Methods of Analysis" some examples for practical purposes are presented below:

Example 1

Determination of protein content in meat products

The general part would include the principle of Kjeldahl's method, e.g. information concerning details of the process of mineralization, the catalyst used, source of heating, etc. would be stated. In this connection the time needed for mineralization should be included as well. Moreover, the process of distillation, the solutions to be uses as well as the method of titration should also be described. As regards the detailed part, the following types of information should be represented: the amount of product to be weighed and the method of expressing results with reference only to the relevant method in the general part.

Example 2

Organoleptic tests of coffee

The general principles for conducting these tests would be described in the general part. However, the detailed part would include the preparation of infusions destined for testing and the interpretation of results only.

METHODS OF ANALYSIS FOR EDIBLE FUNGI AND FUNGUS PRODUCTS, DRIED EDIBLE FUNGI, AND FRESH FUNGUS "CHANTERELLE"

ANALYTICAL CRITERIA	PRODUCT	METHOD OF ANALYSIS ENDORSED	PREPARATION OF TEST SAMPLE
1. Mineral impurities (HCl insoluble)	a) Fresh wild growing fungi	ISO Rec. 763 as amended in CL1970/5 para B	Crush and mix the laboratory sample. Weigh about 10 g.
	b) Fresh cultivated fungi	- " -	- " -
	c) Dried fungi	- " -	- " -
	d) Fungus grits and fungus powder	- " -	Mix the laboratory sample. Weigh about 10 g.
	e) Pickled fungi	- " -	Homogenize the laboratory sample (both solid and liquid phase) and weigh about 20 g.
	f) Fungi in oil 1/	- " -	- " -
	g) Salted fungi	- " -	- " -
	h) Fermented fungi	- " -	- " -
	i) Quick frozen fungi	- " -	After thawing the laboratory sample homogenize both solid and liquid phase. Weigh about 20 g.
	j) Fungus extract and fungus concentrate	- " -	Weigh about 10 g.
	k) Dried fungus concentrate	- " -	- " -
	l) Sterilized fungi	- " -	Homogenize the laboratory sample and weigh about 20 g.
2. Salt content (NaCl)	a) Pickled fungi	JAOAC (March 1971) 32.A01-32.A05	Take for titration 10 ml of liquid phase 2/
	b) Salted fungi	- " -	Take 10 ml of liquid phase, dilute to 100 ml with distilled water. Take for titration 10 ml of the solution 2/
	c) Fermented fungi	- " -	Take 20 ml of liquid phase, dilute to 100 ml with distilled water. Take for titration 10 ml of the solution 2/

1/ Not mentioned in CX/MAS/70/C/5 and not discussed by the Codex Committee on Methods of Analysis and Sampling. To be considered by the Coordinating Committee for Europe.
2/ Preparation of test sample to be reconsidered when the method has been published.

ANALYTICAL CRITERIA	PRODUCT	METHOD OF ANALYSIS ENDORSED	PREPARATION OF TEST SAMPLE
2. (cont.)	d) Fungus extract and fungus concentrate	JAOAC (March 1971) 32.A01-32.A05	Boil 5 g of the product, transfer to a 100 ml measuring flask, dilute to volume with distilled water and filter. Take for analysis 10 ml of the filtrate 2/
	e) Fungi in oil 1/	- " -	2/
	f) Dried fungus concentrate	- " -	Boil 20 g of the product, transfer to a 100 ml measuring flask, dilute to volume with distilled water and filter. Take for analysis 10 ml of the filtrate 2/
	g) Sterilized fungi	- " -	Take for titration 10 ml of liquid phase 2/
3. Water content	a) Dried fungi freeze-dried fungi 1/ dried fungus Shii-ta-ke 1/	AOAC (1965) 29.005	Crush and mix thoroughly the laboratory sample
	b) Fungus grits and fungus powder	- " -	Mix thoroughly the laboratory sample
	c) Dried fungus concentrate	- " -	- " -
4. Acetic acid content	a) Pickled fungi	AOAC (1965) 20.042	Take for titration 10 ml of liquid phase
5. Lactic and/or citric acid 1/ content	a) Fermented fungi	- " -	- " -
	b) Sterilized fungi 1/	- " -	- " -
6. Sugars content	a) Pickled fungi	IFJU No. 4 (1968)	Take for titration the liquid phase

1/ Not mentioned in CX/MAS/70/C/5 and not discussed by the Codex Committee on Methods of Analysis and Sampling. To be considered by the Coordinating Committee for Europe.

2/ Preparation of test sample to be reconsidered when the method has been published.

CONVERSION OF ANALYTICAL RESULTS FROM g/l (mg/l) TO g/kg (mg/kg) AND THE REVERSE

Method of the International Federation of Fruit Juice Producers

In the present collection of IFFJP methods the results are to be stated as g/l or mg/l. Since the standards elaborated express all limits in g/kg or mg/kg, the following conversions are necessary:

(a) For the conversion of g or mg per litre to g or mg per kilogramme the g or mg found in the fruit juice examined are to be divided by the density; the density is to be determined according to IFFJP-Analysis No. 1.

Example

The alcohol content of a fruit juice (density 1.058) found by IFFJP-Analysis No. 2 amounts to 5.3 g/l.

To give the alcohol content in terms of 1 kg fruit juice, the calculation will be:

$$\frac{5.30}{1.058} = 5.01$$

The alcohol content of the above fruit juice thus comes to 5.0 g/kg.

(b) Conversely, to convert g or mg per kilogramme to g or mg per litre, the g or mg found are to be multiplied by the density as given by IFFJP-Analysis No. 1.

Example

In a fruit juice (density 1.065) the tin content was found to be 240 mg/kg. Expressed in terms of 1 litre the tin content will be:

$$240 \cdot 1.065 = 255.6$$

The result will thus be given as:

Tin content: 256 mg/l

Remark

For the examination of beverage samples where the analytical results are to be expressed as g/kg (mg/kg), it is recommended that the samples be weighed out initially rather than be pipetted. The analytical method is otherwise followed as given. The results will then be obtained directly as g/kg (mg/kg), avoiding the need to divide by the density.

Note

It is recommended that in future all the analytical results of international referee methods be expressed as g/kg or mg/kg.

Reference

IFFJP-Analyses General sheet (to be published).

METHOD OF DETERMINATION OF MINIMUM FILL OF CONTAINERS
(endorsed for all Standards for Fruit Juices at Step 8)

Method published in the Alamanac of the Canning, Freezing, Preserving Industries, 55th Edition, 1970 (p. 131-132) E.E. Judge and Sons, Westminster MD (USA):

General Methods for Water Capacity and Fill of Containers

(a) The term "general method for water capacity of containers" means the following method:

(1) In the case of a container with lid attached by double seam, cut out the lid without removing or altering the height of the double seam.

(2) Wash, dry, and weigh the empty containers.

(3) Fill the container with distilled water at 68°F to 1/ 3/16 inch 2/ vertical distance below the top level of the container, and weigh the container thus filled.

(4) Subtract the weight found in (2) from the weight found in (3). The difference shall be considered to be the weight of water required to fill the container.

In the case of a container with lid attached otherwise than by double seam, remove the lid and proceed as directed in clauses (2) to (4) inclusive, except that under clause (3) fill the container to the level of the top thereof.

(b) The term "general method for fill of containers" means the following method:

(1) In the case of a container with lid attached by double seam, cut out the lid without removing or altering the height of the double seam.

(2) Measure the vertical distance from the top level of the container to the top level of the food.

(3) Remove the food from the container; wash, dry, and weigh the container.

(4) Fill the container with water to 3/16 2/ inch vertical distance below the top level of the container. Record the temperature of the water, weigh the container thus filled, and determine the weight of the water by subtracting the weight of the container found in (3).

(5) Maintaining the water at the temperature recorded in (4), draw off water from the container as filled in (4) to the level of the food found in (2), weigh the container with remaining water, and determine the weight of the remaining water by subtracting the weight of the container found in (3).

(6) Divide the weight of water in (5) by the weight of water found in (4), and multiply by 100. The result shall be considered to be the percent of the total capacity of the container occupied by the food.

In the case of a container with lid attached otherwise than by double seam, remove the lid and proceed as directed in clauses (2) to (6) inclusive, except that under clause (4), fill the container to the level of the top thereof.

1/ = 20°C

2/ = approximately 5 mm

DETERMINATION OF TOTAL SOLUBLE SOLIDS CONTENT IN FROZEN FRUITS

(Endorsed for quick frozen Strawberries, quick frozen Peaches and quick frozen Bilberries)

SCOPE

This instruction provides a standardized procedure for determining the total soluble solids content of frozen fruits using the refractometric reading (at 20°C) in terms of the International Sucrose Scale.

The methodology is applicable to both consumer size and bulk containers, although special sampling procedures must be used on large containers.

EQUIPMENT

High speed mechanical blender.

Refractometre, abbe type.

Lens paper, milk straining pads or other suitable filtering medium.

Pliofilm bags or metal containers with tight seal and capacity of about 2 to 3 kilos.

Special sampling devise for bulk containers. Power driven tube of stainless steel or other corrosion resistant metal - diameter 5 to 8 cm, length about 1 metre. One end of tube has serrated edges (like saw teeth) and "set" to prevent binding. A wooden ram or dowel of a slightly smaller diameter will facilitate removal of the core.

REFERENCE TABLES

Temperature Correction Table for refractometric reading for temperatures other than 20°C.

International scale of Refractive Indices of Sucrose Solutions - 1036.

SAMPLING

Retail Size Containers - Use the entire product in the container. Let sample thaw in the original container at room temperature.

Catering Size Containers (Generally of sizes up to 5 kilos or 12 pounds) - Use the entire product if possible. Otherwise let the sample thaw in the original container. Mix the thawed sample thoroughly and remove approximately 1 000 grammes for subsequent analysis.

Bulk Containers - Using the power-driven sampling tube, take three (3) vertical cores evenly spaced around the circumference of the container and one (1) from the centre. Drive sampling tool full length of container, as nearly as possible. Remove cores using the wooden dowel or ram and place in sampling container with tight closure. Combined sample should be at least 1 000 grammes. Let sample thaw in closed sampling container at room temperature.

SOLUBLE SOLIDS DETERMINATION

Mix thawed sample thoroughly in the high speed blender. Generally this will require about two minutes. Filter a portion of the well mixed, blended sample through the filter paper or other medium. Determine the refractometer reading using a drop of the clear, filtered serum. Correct the reading to 20°C and convert to soluble solids using the referenced International Sucrose table.