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# JOINT FAO/WHO CODEX ALIMENTARIUS COMMISSION
## Sixth Session
Geneva, 4 - 14 March 1969

# REPORT OF THE FOURTH SESSION OF THE CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING
Berlin, 11 - 15 November 1968

The Secretariat was requested to edit, complete and check the accuracy of the appendices attached to this report. As far as can be judged in the time available prior to the Sixth Session of the Commission, the appendices are accurate. The Secretariat, however, will wish to re-examine these again after the Commission's session, before any methods are issued with finalized standards for acceptance by Governments.
The following is a key to the abbreviated references to methods:

Ic.R. - Report of the Proceedings of Sessions of ICUMSA
Ic.M. - ICUMSA Methods of Sugar Analysis 1964
C.I.R.F.- Corn Industries Research Foundation
U.S.P. - United States Pharmacopoeia
B.S. - British Standard Institute Method
A.O.C.S.- American Oil Chemists Society
I.O.O.C.- International Olive Oil Council

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# CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING
## Report of the Fourth Session

## PART I

### INTRODUCTION

1. The Codex Committee on Methods of Analysis and Sampling held its fourth session from 11 to 15 November 1968 in Berlin under the chairmanship of Prof. Dr. R. Franck. There were 53 delegates and observers present, representing 18 countries and 13 international organizations. The list of participants appears as Appendix I and the list of documents which the Committee had before it as Appendix II to this Report.

### ADOPTION OF AGENDA

2. The Provisional Agenda was adopted with the deletion of item 10 (c), Determination of TMA, TMAO and TVBN in Fish Muscle, since the standard methods in question were still under consideration by the Codex Committee on Fish and Fishery Products and would not be available before 1969. As regards item 12 (h)*, Inspection Manual for Quick (Deep) Frozen Foods, it was agreed that it should be dealt with particularly in relation to the temperature measurement of quick frozen foods. It was furthermore decided not to bring forward agenda item 11(b), Guidelines and General Principles for the Codex Committee on Methods of Analysis and Sampling, as requested by some delegations, but that the chairman would allow discussion of the respective points thereof whenever appropriate during the session.

### APPOINTMENT OF RAPPORTEUR

3. It was agreed that no rapporteur should be appointed and that the draft report should be prepared by the Secretariat.

## PART II

### DRAFT PROVISIONAL STANDARD FOR HONEY AT STEP 8

4. The Committee had before it the following documents:

- EXEC/68.2/2 (Honey Standard)
- ALINORM 68/23 (Report of the Third Session of the Codex Committee on Methods of Analysis and Sampling)
- MA/68/3, and
- Photostatic copy of a part of the Report of the Fifth Session of the Coordinating Committee for Europe

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*) Note by the Secretariat: This agenda item was not discussed by the Committee.

The discussions were based on the report of the third session of this Committee and the part of the report of the fifth session of the Coordinating Committee for Europe. The Secretariat informed the Committee that the Draft Provisional Standard for Honey had been adopted by the Coordinating Committee for Europe, to be sent through the Secretariat to the Codex Alimentarius Commission for adoption as a provisional standard for the Region of Europe. The Committee discussed fully Section 7 (previous Section 6), Methods of Analysis and Sampling, and agreed upon certain amendments. An amended version of the whole section is attached to this Report as Appendix III.

5. Paragraph 7.1 - Chemical Analysis of Reducing Sugars was adopted with minor modification in the form submitted by the United Kingdom to the Coordinating Committee. The method for preparing the honey sample was somewhat changed according to the AOAC method in order to accommodate dark, high-melting honeys and comb honeys.

6. Concerning the determination of the apparent sucrose content (7.2), the representative of APIMONDIA drew attention to the fact that any melezitose present would also be determined as sucrose by this method. However, the Committee decided to retain the method without an amendment because the respective criterion in the standard was based on the understanding that only the apparent sucrose content would be determined. The Committee agreed that a quantitative method for the determination of the actual sucrose content, when the apparent sucrose content is above 5%, should be evaluated in the future, preferably using chromatographic technique. The delegate of the Netherlands pointed out that modern enzymatic methods should also be considered. A written comment received from the Hungarian delegation regarding the use of methyl-orange for the neutralization after inversion of the sugar solution was noted. In view of the fact that there was not enough information available on the possible effect of the use of this indicator on the results of this determination, the Committee considered that its insertion into the standard was not warranted at this stage.

7. In respect of the Determination of the Moisture Content (7.3) the Wedmore table was amended to read up to 25% moisture content. These additional figures have been proposed by the Coordinating Committee for Europe.

8. The representative of APIMONDIA expressed the opinion that with reference to the determination of acidity (7.6) the AOAC method using a pH-meter would allow the recognition of different honeys, such as honeydew honey. The delegation of Switzerland pointed out that this should be carefully examined and that they themselves would suggest a microscopic method for this purpose which has been found to be very useful with honeys of various origins. The Committee decided to retain its present method because its purpose was simply to ascertain that no acids have been added to the honey.

9. Concerning a description as to how to prepare the soluble starch which is used in the determination of diastase activity (7.7), the delegation of the Netherlands offered to make available to this Committee the agreed process of the BENELUX countries. The Committee decided that

various existing prescriptions for the preparation of the soluble starch should be made available for discussion at the next meeting. The Committee agreed to accept the present method with the understanding that an international reference centre should be established for the supply of such standard reagents as soluble starch. It requested FAO and WHO to examine the possibility of establishing such a centre similar to the ones which are being planned for food colours at present. Concerning the expression and calculation of results in the determination of diastase activity, the representative of APIMONDIA pointed out that the diastase number corresponded closely but not exactly with the Gothe scale number. The Committee decided that the correspondence was sufficiently accurate for the requirements of the standard. Concerning the determination of the diastase activity (7.7) the delegate of the Netherlands stated that the description of this procedure was insufficient and in his opinion may lead to great differences in the results. Before accepting the method it should be described more exactly and in more detail [1]. The Committee repeated its previous opinion that the method for the determination of invertase activity and the use of the diastase/invertase ratio as a measure for the condition of the honey might be considered at a later stage (see Kiermeier, F., and W. Köberlein, (1954) Z. Unters. Lebensmitt., 98, 329).

10. The photometric determination of Hydroxymethylfurfural content (7.8) was endorsed. Considering the use of the reagent p-toluidine (7.8.2.2) it was found advisory to add a footnote containing a warning about the carcinogenic properties of this substance to make certain that analysis would handle the substance with the appropriate care.

11. The Committee decided to endorse the methods of analysis and sampling for honey in their amended form (see Appendix III).

12. The delegate of Canada wished to put on record that, if the present regional draft provisional standard for honey should become a world-wide one, Canada would consider the corresponding AOAC methods, which are part of their legislation, as "equivalent methods". The delegate of the United States expressed a similar opinion. The delegations of Canada and the USA informed the Committee that in many cases the methods used officially in their countries are very similar to those being considered for endorsement at the sessions of this Committee. In many cases these methods are required for use in connection with their laws and regulations. In view of the similarity of some of these methods, they proposed that such methods be considered as equivalent until proved otherwise. They were of the opinion that with such a policy the work of this Committee could be expedited and it would be unnecessary for these countries to make a reservation with regard to acceptance of the endorsed methods. Until such an agreement is reached Canada and the USA must make a general reservation with regard to their acceptance of some of the methods of analysis, indicating that for official purposes they would also have other collaboratively tested international methods which they would regard as equivalent alternatives until proved otherwise.

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[1] The amended determination, giving a more precise description of the method, appears in Appendix III, paragraph 7.

## PART III

## METHODS OF ANALYSIS FOR SUGARS AT STEP 9 AND 8

13. The Committee had before it Appendix III of the Report of its Third Session and document MA/68/14 containing the proposed methods of analysis and sampling for sugars, comments from Governments, and the Report of the Fifth Session of the Codex Committee on Sugars. The delegation of the United Kingdom gave a short summary of the discussion which took place on the methods of analysis at the latter session. The representative of ICUMSA informed the Committee of some matters of procedure for developing ICUMSA methods. He referred to the relationship between ICUMSA and the Codex Alimentarius Commission, pointing out that a special meeting of ICUMSA was held in 1967 for the sole purpose of providing the Codex Committee on Sugars with appropriate methods of analysis. The results of collaborative studies carried out by this Organization, where available, would be submitted in due course. The Committee further expressed its appreciation for the work done by ICUMSA on the methods of analysis for sugars. The Committee considered the proposed methods (see Appendix IX to this Report reproduced from Appendix III to ALINORM 68/23), and the following points arose during the discussion:

### WHITE SUGAR AT STEP 8

14. Polarization "expressed as sucrose": The Committee endorsed the ICUMSA (Ic. R. 1958 p. 84) method with the understanding that polarization was expressed in degrees S since the results obtained with this method did not exactly reflect the sucrose content.

15. Invert Sugar: The Committee endorsed the Berlin Institute method (I.C.M. p. 29) for the determination of invert sugar contents between 0.02% and 0.3% and the LANE and EYNON method (I.C.M. p. 13) for the determination of invert sugar contents above 0.2%. The Committee did not consider it appropriate to endorse the KNIGHT and ALLEN method, which is intended for the determination of invert sugar contents below 0.02% as the lower limit prescribed in the commodity standard is 0.04%.

16. Conductivity Ash: The Committee endorsed the ICUMSA method (Ic. R. 1962, pp. 10-12 and Ic. R. 1966, p. 98).

17. Loss on Drying: The Committee endorsed the ICUMSA method "Determination of Dry Substance c. Atmospheric Pressure Oven" (Ic. M. p. 44) with the understanding that the minimum size of the sample should be 20 g. Several delegations were of the opinion that the determination of the water content by the Karl Fischer method would be preferable to the determination of loss on drying. It was suggested that the view of the Commission should be sought as to the possibility of amending the standard for white sugar and the other sugar standards in this respect at a later date.

18. Colour: The Committee endorsed the ICUMSA method (Ic. M. pp. 57, 58; Ic. R. 1958, p. 52) with the amendment that a membrane filter should be

used instead of the sintered glass filter and that the cell length should be 10 cm (see also Report of the Fifth Session of the Codex Committee on Sugars, paragraph 10(d), ALINORM 69/21). The Committee recommended to ICUMSA that the method be accordingly amended.

19. Sulphur Dioxide: The Committee endorsed the CARRUTHERS, HEANEY and OLDFIELD method (*Int. Sugar J.*, 1965, p. 364).

20. Arsenic: The Committee endorsed the AOAC method (1965) 24.001 (24.016, 24.017).

21. Lead: The Committee endorsed the ICUMSA method (Ic. M. p.48(c)).

22. Copper: The Committee endorsed the ICUMSA method (Ic. M. p.106).

## SOFT SUGARS (WHITE TO DARK BROWN) AT STEP 8

23. Sucrose (Saccharose) and invert sugar expressed as sucrose: The Committee considered the possibility of using a method based on acid inversion and noted that there seemed to be no difficulty to obtain commercially a satisfactory grade of invertase. The Committee endorsed the TATE and LYLE Invertase Method based on the LANE and EYNON method (Ic. M. P.71).

24. Invert Sugar: The Committee endorsed the LANE and EYNON method (without inversion) (Ic. M. p.71).

25. Sulphated Ash: The Committee endorsed the ICUMSA method (Ic. M. p.36).

26. Loss on Drying: The Committee endorsed the ICUMSA method (Ic.M.p.44).

27. Sulphur Dioxide: The Committee considered the proposal of several delegations to replace the method of MONIER WILLIAMS, which, according to the Codex Committee on Sugars, was the one to be used for soft sugars instead of the originally proposed CARRUTHERS, HEANEY and OLDFIELD method, by the MONIER WILLIAMS method modified by TANNER. According to the views expressed by these delegations the TANNER modification was to be preferred. The Committee then decided to endorse the full MONIER WILLIAMS method without the TANNER modification and suggested that collaborative tests be carried out by the countries interested and by ICUMSA using both methods.

28. Arsenic, Lead, Copper: The decisions of the Committee given in paragraphs 20, 21 and 22 apply.

## SOFT WHITE SUGAR AT STEP 8

29. Sucrose (Saccharose) and invert sugar expressed as sucrose: The decision of the Committee given in paragraph 23 applies.

30. Conductivity Ash: The decision of the Committee given in paragraph 16 applies.

31. Colour: The decision of the Committee given in paragraph 18 applies.

## LACTOSE AT STEP 9

32. Lactose Anhydrous: The Committee endorsed the LANE and EYNON method (Ic. M. p.13).

33. Sulphated Ash: The Committee endorsed the ICUMSA Single Sulphation method (Ic. M. p.100).

34. Loss on Drying: The Committee again discussed the possible advantage of determining the true water content of all sugars by means of the Karl Fischer method. It was, however, realized that as these commodity standards were already at Step 8 it would be more appropriate to endorse the method proposed by the Commodity Committee. The Committee therefore endorsed the U.S.P. method (drying 16 h. at 120°C) (U.S.P. 1965, p.336) and decided to delete the reference to the Karl Fischer method.

35. pH (10% solution): The Committee endorsed the ICUMSA method (Ic. M. p.59) and recommended that the concentration of the solution should be amended to 10% by ICUMSA.

36. Arsenic, Lead, Copper: The decisions of the Committee as given in paragraphs 20, 21 and 22 apply.

## GLUCOSE SYRUP, DRIED GLUCOSE SYRUP AT STEP 9

37. Total Solids: The Committee endorsed the CIRF method E 42 "Moisture (Oven filter aid)". The Committee discussed the use of the terms "total solids", "moisture content", "dry matter" and "loss on drying" and recommended that the Commission should consider the possibility of using the same specifications for all sugar standards.

38. Dextrose Equivalent: The Committee endorsed the LANE and EYNON method (Ic. M. p.101).

39. Sulphated Ash: The decision of the Committee as given in para. 27 applies.

40. Sulphur Dioxide: The decisions of the Committee as given in paragraph 27 apply.

41. Arsenic, Lead, Copper: The decisions of the Committee as given in paragraphs 20, 21 and 22 apply.

## DEXTROSE MONOHYDRATE and DEXTROSE ANHYDROUS AT STEP 9

42. Dextrose (expressed as D-glucose): The Committee endorsed the LANE and EYNON method (Ic. M. p.101).

43. Total Solids: The Committee endorsed the ICUMSA method (drying at 100°C for 4 hours reduced pressure) (Ic. M. p.113).

44. Sulphur Dioxide and Sulphated Ash: The decisions of the Committee as given in paragraphs 27 and 33 apply.

45. Arsenic, Lead, Copper: The decisions of the Committee as given in paragraphs 20, 21 and 22 apply.

## METHODS OF SAMPLING OF SUGARS

46. The Committee decided that the ICUMSA methods for

- Sampling from bags (Ic.M.p.80)
- Number of samples to be taken ("$\sqrt[3]{T}$" should read "$3\sqrt{T}$"), and
- Preparation of samples for analysis (Ic. M. p.80)

could not be endorsed as they were not based on modern statistical sampling concepts (see also para. 95). The US delegation indicated that they would be prepared to supply statistical resources for the statistically planned study required.

## PART IV

## METHODS OF ANALYSIS FOR PROCESSED FRUITS AND VEGETABLES AT STEP 9 AND 6 *

47. The Committee had before it Appendix V to the report of its third session and documents MA/68/7 and MA/68/5, containing proposed methods of analysis and comments obtained from Governments. The Committee was informed of the decision of the Codex Committee on Processed Fruits and Vegetables to delete the references to an alternative method for the standard methods for the determination of the drained weight and the alcohol insoluble solids respectively and noted that Canned Sweet Corn had been inadvertently left out from the scope of the drained weight procedure.

48. The Committee endorsed the methods of (a) Determination of Drained Weight for Canned Green Beans, Wax Beans, Peaches, Grapefruit, Asparagus, Pineapple, Green Garden Peas and Sweet Corn, and (b) Determination of Drained Weight for Canned Tomatoes, with the following amendment to (a): "applicable method being the drained weight method for processed fruit and vegetable products of the 'Methods of Analysis of the Association of Official Analytical Chemists (AOAC) 1965, 30.001'." Concerning the Specifications for Circular Sieves in methods (a) and (b) above, the Committee agreed to replace the references to test sieves No. 8 and 2 mesh by reference to 2.8 mm mesh and 11.2 mm mesh ISO test sieves respectively and to give the references to the US sieves as a footnote. Taking into account the results given by the delegation of the USA, the test sieves No. 8 (US) and "No. 7 (2.8 mm)" (nearest corresponding ISO sieve) could be regarded as interchangeable; similarly, 2 mesh (US) and "7/16 inch. (11.2 mm)" (nearest corresponding ISO sieve) can be regarded as interchangeable. The delegation of the USA informed the Committee that in view of the above findings they preferred to continue using the No. 8 and 2 mesh test sieves.

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* See Appendix IV to this Report.

49. Determination of Alcohol-Insoluble Solids (for Canned Green Garden Peas): The Committee endorsed AOAC method (1965), 30.015, with the understanding that it might also be applicable to quick frozen peas with suitable modified sample preparation given in CODEX/ANALYS/68/15 (see paragraph 97). The Committee considered it appropriate that the Joint ECE/Codex Alimentarius Group of Experts on Standardization of Quick Frozen Foods be informed accordingly.

50. Determination of Total Soluble Solids (for Canned Applesauce): The Committee endorsed the AOAC (1965) 29.011 method for the determination of refractive index with the tables 43.008, 43.009 and agreed that the sentence in brackets "(Applicable only to liquid samples containing no undissolved solids)" be deleted and that no correction for water insoluble solids or acidity should be used.

51. Tough String Test (for Canned Green Beans and Canned Wax Beans): The Committee endorsed the method as set out in document MA/68/5. Some delegations, however, expressed the view that the method should be further elaborated before it was to be endorsed as an international referee method.

52. Determination of Calcium (for Canned Tomatoes): The Committee took note of the opinion of the Executive Committee that the determination of calcium could be regarded as being of general application to a number of foods. It was pointed out that the AOAC had developed a specific method for the determination of calcium in canned tomatoes, the suitability of which had also been favourably tested for canned potatoes and canned lima beans. The Committee further noted that there was no ISO Recommendation for a test for calcium salts and that the method given in Appendix III of MA/68/4 was an obsolete one which could not be endorsed, and recommended the method based on JAOAC. The text of the method as set out in JAOAC, (1966) 49, 211 and (1968), 51, 494, is given in Appendix IV to this Report.

53. Syrup Measurements (for Canned Grapefruit, Pineapple, Peaches): The Committee considered the question whether both the refractometric and the hydrometric methods should be endorsed and decided, in view of the limitations of the latter, to endorse only the refractometric method (AOAC, 1965, 29.011, 43.009, 43.008).

54. Howard Mould Count Method (for Canned Tomatoes): The Committee endorsed AOAC method (1965), 36.069 as set out in Appendix IV of document MA/68/4.

55. The Committee endorsed the additional methods of analysis and sampling for processed fruits and vegetables at Step 5 as contained in document MA/68/8 with the sole exception that on page 4 in paragraph (e) "Syrup Measurements" only the refractometric method was retained (see Appendix IV).

## PART V

### METHODS OF ANALYSIS FOR FATS AND OILS AT STEP 8

56. The Committee considered the methods of analysis given in document CODEX/FATS and OILS/40 and the comments received from Governments with a view to selecting one method for each analytical criterion in the various standards for endorsement. It was generally agreed that preference be given to the methods established by IUPAC. The delegations of Australia, Canada and the USA referred to the reservation made by the Canadian and the US delegations as regards the endorsement of the methods of analysis for honey and sugars, and stated that a similar reservation would apply also to the methods of analysis for fats and oils.

### EDIBLE FATS AND OILS AT STEP 8 - GENERAL STANDARD

57. Quality Characteristics

(a) Acid value - The Committee endorsed the IUPAC method (1964), II.D.1.

(b) Peroxide value - The Committee endorsed the IUPAC method (1964), II.D.13, with the understanding that the results were to be expressed as milliequivalent of oxygen per kg of sample.

58. Contaminants

(a) Matter volatile at 105°C - It was mentioned by the representative of AOAC that the use of the vacuum oven method provided results with a better reproducibility. On the other hand the representative of the Olive Oil Council pointed out that while better results were obtained with this method for refined oil, the IUPAC method was to be preferred for virgin olive oil. The Committee endorsed IUPAC method (1964), II.C.1.1 and requested IUPAC to consider the replacement of the obsolete sand-bath by a more appropriate heating device under the section "Apparatus".

(b) Insoluble impurities - The Committee endorsed the IUPAC method (1964), II.C.2.

(c) Soap content - The Committee endorsed the method BS 684: 1958, page 49.

(d) Iron - The Committee endorsed the method BS 684: 1958, page 92.

(e) Copper - The Committee endorsed the AOAC method (1965) 24.023.

(f) Lead - The Committee endorsed the AOAC method (1965) 24.053.

(g) The methods for trace elements specified in (d), (e) and (f) above might in due course be replaced by atomic absorption spectroscopy.

(h) Arsenic - The Committee endorsed the AOAC method (1965) 24.011 (24.016, 24.017).

59. Identity Characteristics

(a) The Committee noted that according to the views expressed by the Codex Committee on Fats and Oils at its Fifth Session, gas chromatographic methods would be used to an increasing extent replacing the classical methods for identification of fats and oils.

(b) The Committee discussed the possibility of replacing the classical methods of identification of fats and oils by gas chromatographic methods. The delegation of the U.K. drew the attention of the Committee to the views expressed by the Codex Committee on Fats and Oils that the replacement of the classical methods would probably proceed in three stages:

(i) Use of the classical methods;
(ii) use of both the classical and gas chromatographic methods, and collecting data derived from the latter, which would be used along with the classical values;
(iii) classical parameter becoming superseded by the gas chromatographic ones.

(c) Relative density * - The Committee decided to endorse for the time being BS 684: 1958, page 10, method 1 (see Appendix VIII) and noted the reservation of the delegation of Portugal that the reference temperature of all oils should be 20°C (see also section Olive Oil of this Report, paras. 74-77).

(d) Refractive Index - The Committee endorsed the IUPAC method (1964), II.B.2 and noted that the delegate of Portugal made the same reservation as stated for the determination of the relative density.

(e) Saponification Value - The Committee endorsed the IUPAC method (1964), II.D.2.

(f) Unsaponifiable Matter - The Committee discussed the advantages of using diethyl ether instead of light petroleum for the extraction and endorsed IUPAC method (1964), II.D.5.3 (diethyl ether) taking into account that certain tolerances given in the standards were evidently based on this method and that the diethyl ether extract could be used for further tests. It was, however, pointed out by several delegations that in the case of fats and oils with low contents of unsaponifiable matter the extraction should be carried out with petroleum ether.

(g) Iodine Value (Wijs) - The Committee endorsed the IUPAC method (1964), II.D.7.3. Concerning the necessary excess of iodine, the Committee concluded that IUPAC should be asked to examine the other methods in the document.

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* Note by the Secretariat: The method given as Appendix VIII has been amended as follows: (a) "relative density" replaced by the I.S. term "specific gravity", (b) reference temperature 20°C in place of 15.5°C, (c) the general wording "fats and oils" used instead of "oils".

(h) Crismer Value - The Committee concluded that the AOCS method Cb 4-35 could be endorsed for rapeseed oil only and removed from the general methods of analysis for edible oils.

60. "Specific" Tests

(a) Arachis Oil: Determination of arachidic acid and higher fatty acid content by either the Modified Renard Test or the Arachis Oil Test (Evers). The Committee noted that the determination of arachidic acid might be replaced by the gas chromatographic method and that some delegations were in favour of adding the Bellier Index to the Arachis Oil Standard. The Committee endorsed the AOAC method (1965) 26.077 and BS 684: 1958, page 97, noting that these tests were useful for the determination of peanut oil in olive oil, cottonseed oil, corn oil and soyabean oil, but not suitable for the evaluation of the purity of peanut oil. The Committee expressed the view that the heading "specific tests" was misleading and should be replaced by "specific identification tests".

(b) Cottonseed Oil: Halphen Test. The Committee endorsed the AOCS official method Cb 1-25 and noted that the method might be replaced by gas chromatographic method in due course.

(c) Sesameseed Oil: Modified Villavecchia Test and Sesame Oil Test (Baudouin). The Committee was informed that sesameseed oil might get oxidized after long storage and that the tests are likely to be disturbed. It was noted that the Modified Villavecchia Test could not be used in refined sesame oils. It endorsed the AOCS official method Cb 2-40 and the method BS 684: 1958, page 96. The Committee considered it useful to incorporate the above remarks in the standard.

## LARD AND RENDERED PORK FAT AT STEP 8

61. Quality Characteristics

(a) Acid Value )
(b) Peroxide Value ) The decisions of the Committee as given in paragraph 57(a) and (b) apply.

62. Contaminants

(a) Matter volatile at 105°C )
(b) Insoluble impurities )
(c) Soap content )
(d) Iron )
(e) Copper )
(f) Lead )
(g) Arsenic )
The decisions of the Committee as given in paragraph 58(a) to (h) apply.

63. Identity Characteristics

(a) Relative density )
(b) Refractive index *) ) The decisions of the
(c) Saponification value ) Committee as given in
(d) Unsaponifiable matter ) paragraph 59(c) to (g)
(e) Iodine value (Wijs) ) apply.
(f) Titre - The Committee endorsed the IUPAC method (1964) II.B.3.2.
(g) "Bömer Value" - The Committee considered the proposal to include the Bömer Value in the Standard and pointed out that the ISO and DGF (Deutsche Gesellschaft für Fettwissenschaft) had agreed on the method, and that the Codex Committee on Fats and Oils be asked to consider this matter. 1/

*) The Portuguese delegation drew the attention of the Committee to the fact that it was not possible to determine the refractive index of some samples of lard and edible tallow at the reference temperature of 40°C, as they were not liquid or transparent at this temperature. A higher temperature should be envisaged.

## PREMIER JUS AND EDIBLE TALLOW AT STEP 8

64. Quality Characteristics

(a) Acid Value ) The decisions of the Committee as given
(b) Peroxide Value ) in paragraph 57(a) and (b) apply.

65. Contaminants

(a) Matter volatile at 105°C )
(b) Insoluble impurities ) The decisions of the
(c) Soap content ) Committee as given in
(d) Iron ) paragraph 58(a) to (h)
(e) Copper ) apply.
(f) Lead )
(g) Arsenic )

66. Identity Characteristics

(a) Relative density )
(b) Refractive index ) The decisions of the
(c) Saponification value ) Committee as given in
(d) Unsaponifiable matter ) paragraphs 59(c) to (g)
(e) Iodine value (Wijs) ) and 63(f) apply.
(f) Titre )

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1/ Note by the Secretariat: The document ISO/TC34/SC6/WG3 (Secr.-37) 61 on "Bömer value" is still under consideration by ISO.

## MARGARINE AT STEP 8

67. Total Fat Content - The Committee considered the question whether a direct or an indirect method would be preferable and endorsed the official Netherlands Food Analysis Method as given in Appendix I of document CODEX/FATS AND OILS/40. The Committee decided to delete the references to the determination of the water content and the non-fat residue from the list of methods. It noted the reservation of the delegation of the Federal Republic of Germany, namely that the method endorsed be replaced by a more appropriate one. It also noted the reservation of the delegation of the USA indicating that the use of the AOAC method 15.133 (direct determination) was preferred.

68. Milk Fat Content

(a) Reichert (R) value, (b) Polenske (P) value, (c) Kirschner (K) value

The Committee noted that both the methods of IUPAC and AOAC were necessary because of the extremely empirical nature of the methods depending on regional deviations and endorsed the following methods:

for (a) and (b): IUPAC method (1964) II.D.9 and AOAC methods (1965, 26.032, 26.033

for (c): BS 684: 1958, page 70

with the understanding that the methods might be very soon replaced by gas chromatographic methods. The Committee also considered the determination of the butyric acid value to be a better means for the evaluation of the milk fat content.

69. Vitamin A - The Committee endorsed the AOAC method (1965) 39.001-7 with the understanding that the results be expressed as retinol (Vit. A-alcohol) in microgrammes.

70. Vitamin D - The Committee endorsed the AOAC method (1965) 39.115-29.

71. Vitamin E - The Committee endorsed the method as described in Analyst, (1959), 84, 356.

72. Sodium Chloride - The Committee endorsed FAO/WHO Standard B.8 "Determination of the Salt (Sodium chloride) Content of Butter" 1/ for the determination of the sodium chloride content of margarine and agreed that a potentiometric method should be developed for the determination of NaCl contents in margarine below 1.0%.

73. Edible carbohydrate sweetening matters, edible proteins - The Committee agreed that the references to the methods given be deleted as they were not specifically drawn up for the determination of sugars and edible proteins respectively in margarine and that the absence of these methods would not impede the advancement of the standard.

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1/ Joint FAO/WHO Committee of Government Experts on the Code of Principles concerning Milk and Milk Products, Report of the 10th Session (1967) (SP 10/105 - 10th, Appendix IV-D)

## OLIVE OIL AT STEP 5

74. The representative of the Olive Oil Council informed the Committee that his Organization consisted of the ten leading olive oil producing countries, representing 99.1% of the world exports for this product. He explained that the work on methods of analysis was undertaken in close cooperation between IUPAC, ISO and IOOC; methods considered include gas chromatography and UV-spectrophotometry. These two methods were regarded as suitable for olive oils, having been collaboratively tested by 23 laboratories in different countries.

75. Fatty Acid Composition - The Committee discussed the question of including the method of IUPAC II.D.9 (to be published) for the determination of fatty acid composition of olive oil by gas chromatography. As the Committee did not have this method before it, and in view of the fact that the olive oil standard is at Step 5, it decided to postpone the adoption of this method for olive oil stating, however, that it agreed with this method in principle.

76. The Committee endorsed the following methods of analysis for olive oils:

| | |
|---|---|
| (a) Relative Density at 20°C | IUPAC (1954) p. 37 |
| (b) Refractive Index at 20°C | IUPAC (1964) II.B.2 |
| (c) Iodine Value (Wijs method) | IUPAC (1964) II.D.7.3 |
| (d) Saponification Value | IUPAC (1964) II.D.2 |
| (e) Unsaponifiable Matter | IUPAC (1964) II.D.5.2 |
| (f) Bellier-Index | The methods are described in the corresponding Appendices 2, 3, 4, 5, 6 and 7 of CODEX/FATS AND OILS/40 |
| (g) Semi-siccative Oils Test | |
| (h) Residue Olive Oil Test | |
| (i) Cottonseed Oil Test | |
| (j) Tea Oil Test | |
| (k) Sesame Oil Tests | |
| (l) Acidity | IUPAC (1964) II.D.1 |
| (m) Peroxide Value | IUPAC (1964) II.D.13 |
| (n) Specific Extinction in U.V. | The method is described in Appendix 8 of CODEX/FATS AND OILS/40 |
| (o) Moisture and Volatile Matter | IUPAC (1964) II.C.1.1 |
| (p) Insoluble Impurities | IUPAC (1964) II.C.2 |
| (q) Soap Test | The method is described in Appendix 9 of CODEX/FATS AND OILS/40 |

77. The Committee, also considering that the Tea Oil Test would need to be improved in order to avoid possible interference with some pure olive oils, recommended to IOOC to undertake a study of this matter.

## PART VI

## METHODS OF ANALYSIS FOR COCOA PRODUCTS AND CHOCOLATE AT STEP 4

78. The delegate of Canada informed the Committee that at its last meeting the Codex Committee on Cocoa Products and Chocolate had selected and recommended certain methods of analysis. These methods which are mentioned in the Report of that Committee (ALINORM 69/10) should be

reproduced and put on the agenda for the next meeting of the Codex Committee on Methods of Analysis and Sampling.

## PART VII

## METHODS OF ANALYSIS FOR FRUIT JUICES AT STEP 5

79. The ECE/Codex Alimentarius Group on Fruit Juices will have before it at its next session a synopsis of government comments on various methods of analysis and will then select appropriate methods of analysis. The Committee was of the opinion that when selecting methods of analysis for consideration by the Codex Committee on Methods of Analysis and Sampling, the Group should take into account the Guidelines and General Principles for the Codex Committee on Methods of Analysis and Sampling, as adopted by this Committee (see Appendix V).

## PART VIII

## PROPOSED SAMPLING PLANS FOR THE QUALITY ASSESSMENT OF PROCESSED FRUITS AND VEGETABLES AT STEP 5

80. The Committee based its discussions on document ALINORM 69/27 containing the revised version of the sampling plans discussed at the fifth session of the Codex Committee on Processed Fruits and Vegetables. The following points were raised:

(a) It was pointed out that according to the amended 'scope' section the Sampling Plans were no longer applicable to lots at retail level and to factors which might present a hazard to the health of the consumer.

(b) The Committee agreed to endorse the Sampling Plans for the quality assessment of processed fruits and vegetables, noting that they applied only to the assessment of the quality factors (mainly visible defects) laid down in the standards concerned.

(c) The representative of the European Organization for Quality Control (EOQC) expressed some doubt as to the validity of the sampling plans contained in the document because in his opinion they were no longer in line with the new internationally accepted principles for the elaboration of sampling plans for "inspection by attributes".

(d) The Committee had also before it document MA/68/6 containing comments by the Japanese Government. The document had been received too late to be considered by the Committee but it was hoped that the comments would be considered at a later stage.

(e) The representatives of ISO and EOQC drew attention to the fact that many other aspects of sampling remain to be covered, in particular statistical sampling plans needed for various types of criteria and for various types of products.

(f) The delegate of the U.K. informed the Committee about the work of ISO in progress in the wide field of sampling carried out by a working group of TC34. He also mentioned the previous work which ISO had made available to the Codex Alimentarius Commission in a document (ALINORM 65/25(1)) setting out the general approach to and principles for food sampling as well as the mathematical basis for sampling procedures. He emphasized the need to differentiate clearly the particular purpose of the sampling for which a method was to be drawn up and listed a number of these purposes. The Committee was unanimous in emphasizing the importance it attached to the elaboration of comprehensive sampling plans and procedures and stressed the urgency of this work in connection with the elaboration of Codex standards. The Committee wished to draw attention to the importance which it attached to the work in this field and requested ISO to proceed with the tasks in question with all possible spped.

(g) The delegate of Canada suggested that the Secretariat of the Joint FAO/WHO Codex Alimentarius Commission should provide for the assistance of a consultant who is a specialist on this subject and who would collect and evaluate all available relevant information on the subject of food sampling and prepare working papers for this Committee in the near future. These papers would also be made available to ISO/TC 34 for consideration. The Committee agreed to request the Secretariat to examine the possibilities of carrying out this proposal and also expressed its gratitude to ISO for their offer of cooperation.

## TECHNICAL PROCEDURE OF SAMPLING FOODS AT STEP 5

81. The Committee had before it the documents ALINORM 68/23, Appendix IX, containing the revised version of the draft provisional standard for the Technical Procedure of Sampling Foods, which had been submitted to governments for comment at Step 3, CODEX/ANALYS/68/6-1 containing US comments on the above paper, MA/68/7 containing a note on sampling by the Secretariat and CODEX/ANALYS/68/18 containing comments by the Government of Argentina. The Committee agreed to adopt the above Draft Provisional Standard at Step 4 of the Procedure for the Elaboration of World-wide Codex Standards with the amendments proposed in the US comments: i.e. in paragraph 2.1.1 the words "or sworn independent" were eliminated and in paragraph 2.1.3 the respective passage was changed to read: "signed by an authorized sampling agent and countersigned by any witnesses present, if necessary". An amended version of the standard is attached to this Report as Appendix VI.

82. The question raised in document MA/68/7 concerning technical procedures for sampling various groups of foods of similar consistency was briefly discussed. It was considered that this might prove difficult. The Committee decided, however, that the pertinent section of Standard B.1 contained in the Sixth Edition of the Code of Principles concerning Milk and Milk Products and Associated Standards should be brought to the attention of Codex Commodity Committees in order that they may examine whether the sampling procedures could be applied to their commodities.

The relevant sections are:

Standard No. B.1 (1966)
- Section 3: Sampling of milk and liquid milk products except evaporated and sweetened condensed milk
- Section 4: Sampling of condensed milk and evaporated milk
- Section 5: Sampling of dried milk and dried milk products
- Section 6: Sampling of butter
- Section 7: Sampling of cheese

83. The Committee was informed that the International Committee on Microbiological Specifications for Foods, a standing committee of the International Association of Microbiological Societies, is working on the subject of food bacteriology and is dealing now with procedures and sampling plans for bacteriological examination. It was understood that any such methods would replace the present appropriate section of the draft standard, as they become available.

## PART IX

### STANDARD LAYOUT

84. The delegate of the United Kingdom introduced the (Revised) STANDARD LAYOUT FOR A CODEX METHOD OF FOOD ANALYSIS and the NOTES contained in CODEX/ANALYS/68/8, which were prepared by the UK delegation on the request of the Codex Committee on Methods of Analysis and Sampling. The UK delegate drew the Committee's attention to some items which had been incorporated into the layout, namely: Section 3, Field of application; Section 5, Principle of methods, Reactions, Specificity; and Section 10, Calculation, Expression and Interpretation of Results, in order to highlight the importance of the subjects concerned. He mentioned that full use of the draft revision of ISO R 78 had been made when the document was prepared and that both ISO Technical Committee TC/34 Agricultural Food Products and TC/47 "Chemistry" recently welcomed the document. He explained that it was to be considered by the ISO/TC 78 Working Group early in the New Year, after which it was hoped to become an ISO Recommendation within about one year.

85. The Committee considered the layout and the notes on the use of the standard layout and on the individual subject sections and agreed it was a highly useful document and it decided to adopt it in toto and to recommend it for use by the Commodity Committees; it decided also to invite Government comments so that its contents could be reviewed from time to time. The Committee considered it desirable to prepare the French and Spanish versions of this document (see Appendix VII).

## PART X

## METHODS FOR THE DETECTION AND IDENTIFICATION OF COLOURS ADDED TO FOOD

86. The Committee briefly discussed the working paper (CODEX/ANALYS/68/9) and provisional general scheme for the extraction and identification of synthetic water-soluble Dyes from Foods (Appendix I) and decided to send it out to Governments for comments at Step 3. The delegate of the Netherlands mentioned that a similar method had been agreed upon in BENELUX and that it was hoped to have this adopted by the EEC soon.

87. The Committee expressed its gratitude to the UK delegation for their work and requested that all material on this subject together with other comments should be sent to the UK delegation as soon as possible.

88. With reference to the list of food colours dealt with in the above document some delegations were of the opinion that it should be reduced to the permitted colours contained in Codex standards and in a list by the Codex Committee on Food Additives, including categories A, B, CI and CII (see ALINORM 68/12, Appendix XI). It was mentioned that methods for prohibited food colours would also be necessary for control purposes.

89. The Committee requested the Secretariat to prepare a list of food colours which should be kept in the planned international reference centres.

## PART XI

## GUIDELINES AND GENERAL PRINCIPLES FOR THE CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

90. The Committee had before it document MA/68/10 containing references to certain recommendations and decisions of the Executive Committee (ALINORM 69/3, paragraphs 3-8). In the Report of the Second Session of the Codex Alimentarius Commission (1964) certain principles for the guidance of the Codex Committee on Methods of Analysis and Sampling had been laid down. The Executive Committee considered some of these as obsolete and others in need of revision. The majority of the Committee on Methods of Analysis and Sampling was of the opinion that it would be advisable to have only one referee method. It was pointed out, however, that this could have certain legal implications and that the possibility of having alternative methods should remain open as agreed upon previously. Some delegations were of the opinion that a clause allowing the use of methods other than referee methods, if mutually agreed upon by the parties concerned, should be added. Where Codex standards contained numerical parameters based on certain methods, no alternative methods would be possible. The Committee restated its previous decision that any "alternative method" should be endorsed by the Codex Committee on Methods of Analysis and Sampling. Some other changes of the paper included rearranging of the subparagraphs according to their importance and a qualification regarding the degree of accuracy needed. The amended version is attached to this Report as Appendix V.

91. Document MA/68/10 also contained some other proposals by the Secretariat regarding procedure on the basis of the decisions of the Executive Committee at its Twelfth Session. The Committee took note of the decisions concerning methods of analysis proposed by Codex commodity committees and methods of analysis generally applicable to a number of foods (see ALINORM 69/3, para. 4), and it agreed with them.

92. Methods of analysis of food additives as such (exclusive of pesticides) (see ALINORM 69/3, para. 7) - The Committee agreed with this change of procedure but recommended at the same time that the methods as elaborated by the Joint FAO/WHO Expert Committee on Food Additives would not necessarily have to be put through the Procedure for the Elaboration of Codex Standards. It was understood that the Committee on Methods of Analysis and Sampling would proceed with its work on the determination of additives in foods.

93. The Committee agreed with the proposed change of procedure concerning methods of analysis of pesticide residues in food (see ALINORM 69/3, para. 8)

94. Microbiological methods of analysis and sampling (see ALINORM 69/3, para. 6) - The Committee took note of the recommendation of the Executive Committee concerning microbiological methods of analysis and sampling and pointed out that there were certain methods of analysis of this kind which should be referred to the Committee on Methods of Analysis and Sampling, such as chemical methods for toxins formed by microorganisms, the use of chemical and physical methods for the determination of certain microorganisms which are of no great concern from a health point of view (e.g. Howard mould count), the use of biological test methods for compositional criteria (e.g. vitamins, enzymatic tests), etc. The Committee agreed that there might be other aspects where overlapping was unavoidable and the above recommendation should be clarified before a final position could be taken by the Committee.

## PART XII

## GENERAL PART OF THE SECTION "METHODS OF ANALYSIS" OF THE CODEX ALIMENTARIUS

95. In view of the fact that no working paper had been received on this subject, it was decided to postpone it until the next meeting.

## FUTURE WORK

96. The Committee had before it the documents listed below, but owing to lack of time, considered them only briefly.

- MA/68/2: Uniformity of Methods of Sensoric Analysis
- CODEX/ANALYS/68/12: Enzymatic analysis of sugars in honey
- CODEX/ANALYS/68/13: Spectrophotometric determination of HMF content of honey
- MA/68/11: Methods of analysis for preservatives in food
- MA/68/12: Methods of analysis for antioxidants in food
- MA/68/13: Methods of analysis referred to Codex commodity committees (Inspection Manual for Quick Frozen Foods)

It was generally agreed that these items would be reconsidered at the next session. As regards the microscopic examination of honey (pollen analysis)(CODEX/ANALYS/68/10) and the GLC determination of sugars in honey (CODEX/ANALYS/68/11), the Committee noted that these methods might not be necessary for the honey standard at the present time. The Committee agreed, however, that the above methods were of sufficient importance for the honey standard and could be included therein after the merits of the pollen analysis has been evaluated and the procedure of the GLC method adequately described. The Committee agreed that these two methods should be sent to Governments for comments.

97. As a matter of routine, the documents CODEX/ANALYS/68/15 "Determination of the alcohol-insoluble solids content of Quick Frozen Peas" and CODEX/ANALYS/68/14 "Net Weight Determination of Frozen Fruits and Vegetables" will be sent to Governments for comment.

98. The delegate of Argentina expressed the reservations of his country as regards the decisions of this Committee which were not in agreement with the comments from the Argentine Republic in the document CODEX/ANALYS/68/18.

## OTHER BUSINESS

99. It was suggested that the Codex Alimentarius Commission establish a small group for drawing up a plan for the uniform numbering and layout of all Codex documents. The Committee agreed to this proposal.

## DATE OF NEXT SESSION

100. The chairman stated that the next meeting would be held some time in October 1969. The US delegate pointed out that the Annual Conference of the AOAC would be held from 13th to 18th October 1969, and the chairman agreed to take this into consideration. A delegation suggested that in view of the rather heavy agenda which the Committee would have to deal with at its next session, prolonging the session over ten days should be considered.

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APPENDIX I

# LIST OF PARTICIPANTS

ALINORM 69/23

APPENDIX II

# DOCUMENTS AND LITERATURE REFERENCES TO METHODS OF ANALYSIS AND SAMPLING

| Document No. | Agenda Item No. | Subject | Language (*) |
|---|---|---|---|
| ALINORM 68/23 | all items | Report of the Third Session of the Codex Committee on Methods of Analysis and Sampling | E,F,S |
| MA/68/1 | 11 (a) | General Part of the Section "Methods of Analysis" of the Codex Alimentarius (Proposal of Poland) | E,F,S |
| MA/68/2 | 12 (a) | Unification of Methods of Sensoric Analysis (Proposal of Poland) | E,F,S |
| EXEC/68.2/2 | 4 | Draft Provisional Standard for Honey | E |
| MA/68/3 | 4 (a) | Comments on the Methods of Analysis section of the Draft Provisional Standard for Honey | E,F |
| MA/68/3, Addendum 1 | 4 (a) | Comments of the Netherlands | E,F |
| MA/68/3, Addendum 2 | 4 (a) | Comments of Hungary | E,F |
| MA/68/3, Addendum 2 - CORRECTION *) | 4 (a) | CORRECTION regarding MA/68/3 - Addendum 2 | E |
| CODEX/EURO/68/2 | 4 | Further Comments on the Draft Provisional Standard for Honey | E |
| CODEX/EURO/68/2, Addendum 1 | 4 | Comments of the Netherlands | E,F |
| CODEX/EURO/68/2, Addendum | 4 | Comments of Australia | E,F |

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(*) Key E = English
F = French
S = Spanish

| Document No. | Agenda Item No. | Subject | Language |
|---|---|---|---|
| MA/68/4 | 6 (a) | Methods of Analysis for Processed Fruits and Vegetables, Note by the FAO Secretariat | E,F |
| MA/68/4, Appendices IV, V and VI | 6 (a) | Official Methods of Analysis, A.O.A.C. | E |
| MA/68/5 | 6 (b) | Tough String Test | E,F |
| MA/68/6 | 8 (a) | Proposed Sampling Plans for the Quality Assessment of Processed Fruits and Vegetables | E,F |
| ALINORM 69/27 | 8 (a) | Sampling Plans for Processed Fruits and Vegetables (Revised Draft Standard prepared by USA) | E |
| MA/68/7 | 8 (d) | Note on Sampling by the Secretariat | E,F |
| MA/68/8 | 10 (a) | Methods of Analysis in Codex Standards which will be presented to the Codex Alimentarius Commission at Step 5 | E,F |
| MA/68/9 | 10 (c) | Determination of TMA, TMAO and TVBN in Fish Muscle (Note by the Secretariat) | E,F |
| MA/68/10 | 11 (b) | Guidelines and General Principles for the Establishment of Codex Methods of Analysis | E,F |
| MA/68/11 | 12 (f) | Methods of Analysis for Preservatives | E,F |
| MA/68/12 | 12 (g) | Methods of Analysis for Antioxidants | E,F |
| MA/68/13 | 12 (h) | Methods of Analysis referred by Codex Commodity Committees | E,F |
| MA/68/14 | 5 | Methods of Analysis for Sugars | E |
| MA/68/14-Corr | 5 | Corrigendum | E |
| ALINORM 69/21 | 5 | Report of 5th Session of the Codex Committee on Sugars, 10 - 12 September 1968. | E,F,S |

| Document No. | Agenda Item No. | Subject | Language |
|---|---|---|---|
| CODEX/ANALYS/68/1 | | Provisional Agenda | E |
| CODEX/ANALYS/68/2-1 | 5 (a) | U.S. Comments on Methods of Analysis for Sugars | E,F |
| CODEX/ANALYS/68/2-2 | 5(a)+(b) | Comments of the Netherlands and Sweden on the proposed Methods of Analysis for Sugars | E |
| CODEX/ANALYS/68/3-1 | 5 (b) | U.S. Comments on Methods of Analysis for White Sugars | E,F |
| CODEX/ANALYS/68/3-2 | 5 (b) | Comments of Cuba | E,F,S |
| CODEX/ANALYS/68/4-1 | 6 | U.S. Comments on Methods of Analysis for Processed Fruit and Vegetables | E,F |
| CODEX/ANALYS/68/5-1 | 7 | U.S. Comments on Methods of Analysis for Edible Oils | E,F |
| CODEX/ANALYS/68/5-2 | 7 | Comments of the Netherlands | E,F |
| CODEX/ANALYS/68/5-3 | 7 | Comments of Sweden | E |
| CODEX/FATS AND OILS/40 | 7 | Methods of Analysis, submitted by the Codex Committee on Fats and Oils (1967) | E |
| CODEX/FATS AND OILS/ *) REPORT V | 7 | Report of Fifth Session of the Codex Committee on Fats and Oils, 16-20 September 1968 | E,F |
| CODEX/ANALYS/68/6-1 | 8 (b) | U.S. Comments on Draft Provisional Standard for the Technical Procedure of Sampling Foods | E,F |
| File Code: 120-A-1 *) | 8 (b) | Sampling Procedures - General Instructions, for use of USDA Processed Foods Inspectors, October 1960, U.S. Dept. of Agriculture, Washington, D.C. | E |
| CODEX/ANALYS/68/7 | 8 (c) | Note on Sampling, prepared by the Canadian Delegation | E,F |
| CODEX/ANALYS/68/8 | 9 | Explanatory Notes for the Standard Layout for a Standard Method of Food Analysis, prepared by the U.K. Delegation | E,F |

| Document No. | Agenda Item No. | Subject | Language |
|---|---|---|---|
| CODEX/ANALYS/68/9 | 10 (b) | Methods for the Detection and Identification of Colours added to Food, submitted by the U.K. Delegation | E,F |
| CODEX/ANALYS/68/10 | 12 (b) | Microscopical Analysis of Honey, prepared by the International Commission for Bee Botany of IUBS | E,F,G |
| CODEX/ANALYS/68/10 | 12 (c) | Comments on Chemical Methods for the Evaluation of Sugars in Honey, submitted by APIMONDIA | E,F |
| CODEX/ANALYS/68/13 | 12 (e) | U.V.-spectrophotometric Determination of Hydroxymethylfurfural in Honey according to O. Winkler, submitted by the Netherlands' Delegation | E,F |
| CODEX/ANALYS/68/14*) | 12 (h) | Net Weight Determination of Frozen Fruits and Vegetables, submitted by the U.S. Delegation | E |
| CODEX/ANALYS/68/15*) | 12 (h) | Determination of the Alcohol insoluble solids Content of Quick Frozen Peas, submitted by the U.S. Delegation | E |
| AGRI/WP.1/523 | 12 (h) | Inspection Manual for Quick (Deep) Frozen Foods, proposed by the USA | E |
| CODEX/ANALYS/68/16*) | 4 (a) | Table for the Estimation of Moisture Content, proposed by the FAO Food Standards Program, Rome, regarding the Draft Provisional Standard for Honey, Doc. EXEC/68.2/2, para. 6.3 | E |
| CODEX/ANALYS/68/17*) | 4 (a) | Extract of Comments made by The New Zealand Department of Agriculture on Doc. EXEC/68.2/2 | E |
| CODEX/ANALYS/68-Doc.*) | | List of Documents | E |

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*) distributed during the session

| Document No. | Agenda Item No. | Subject | Language |
|---|---|---|---|
| CODEX/ANALYS/68/18*) | various | Observations of Argentina regarding various items | F |
| CODEX/ANALYS/68/19*) | 7 | Comments of the Federal Republic of Germany on Methods of Analysis for Edible Oils. | E |
| MA/68/3-Addendum 3*) | 4(a) | Comments of New Zealand on the Methods of Analysis Section of the Draft Provisional Standard for Honey | E |
| MA/68/6 Synopsis *) Addendum | 8(a) | Comments by Japan on the Draft Sampling Plans for Processed Fruits and Vegetable | E |
| MA/68/14 *) Addendum 1 | 5 | Methods of Analysis for Sugars | E |
| Extract from ALINORM 69/21 *) | 5 | Extract from the Report of the 5th Session of the Codex Committee on Sugars | E |
| without reference number *) | 4 | Re-Draft of Section 6.1 of the Draft Provisional Standard for Honey, submitted by the United Kingdom, as corrected version, accepted by the Coordinating Committee. | E |

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*) Distributed during the session

## LITERATURE REFERRED TO IN THE ABOVE DOCUMENTS

### 1. General

- Official Methods of Analysis of the Association of Official Agricultural Chemists (9th ed. 1960 and 10th ed. 1965) A.O.A.C., P.O.B. 540, Benjamin Franklin Station, Washington 4, D.C.
- Journal of the Association of Official Analytical Chemists (J.A.O.A.C.) (Baltimore)
- Draft ISO Recommendations (Nos. 904, 1223, 1224, 1226) Address: 1, rue de Varembé, 1211 Geneva, 20 Switzerland

### 2. Sugars

INTERNATIONAL COMMISSION FOR UNIFORM METHODS OF SUGAR ANALYSIS (I.C.U.M.S.A.)

- Report of the Proceedings of the Twelth Session held at the National Bureau of Standards and at the Shoreham Hotel, Washington D.C., U.S.A., from 2 to 6 June, 1958.

  I.C.U.M.S.A. address: 23, Avenue d'Iena, Paris 16ème, France

INTERNATIONAL COMMISSION FOR UNIFORM METHODS OF SUGAR ANALYSIS (I.C.U.M.S.A.)

- Report of the Proceedings of the Thirteenth Session held at the University of Hamburg and at The Atlantic Hotel, Hamburg, Germany, from 26 to 31 August, 1962.

INTERNATIONAL COMMISSION FOR UNIFORM METHODS OF SUGAR ANALYSIS (I.C.U.M.S.A.)

- ICUMSA Methods of sugar analysis, H.C.S. De Whalley, Amsterdam. Elsevier 1964
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- Angewandte Chemie (Weinheim, Germany), 1935 (Loss on drying)
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- U.S. Pharmacopoeia, 17th Revn. (New York), 1965 (Loss on drying) pp. 336, 909 and 924
- C.I.R.F. Standard Analytical Methods of the Member Companies of the Corn Industries Research Foundation Inc. prepared by the Analytical Procedures Sub-Committee of the Technical Advisory Committee, First Ed. - 1001, Connecticut Ave., Washington 6, D.C. Method E. 42 (Total Solids in Syrup), 1962.

## 3. Oils

- I.U.P.A.C. Analysis of oils and fats, 4th edition, 1954. Société d'Edition d'Enseignement Supérieur, 5, Place de la Sorbonne, Paris (5ème)
- Standard Methods of the Oils and Fats Section of the I.U.P.A.C., 5th Edition, incorporating First Supplement up-to-date to 1965, Butterworths, London, 1966.
- Supplement 1966 to the above.
- British Standard 684: 1958 (UDC 665.014: 543) Methods of Analysis of Oils and Fats, British Standards House, 2, Park St., London, W.1
- Analyst (Cambridge, England), 1959 (vitamin E)
- American Oil Chemists Society, (A.O.C.S.) Official and tentative methods of the American oil chemists society, 2nd ed. Chicago, Ill. 1957 including additions and revisions 1947. Editor of Analytical Methods 1943-1950 : V.C. Mehlenbacher; 1950-1958 : T.H. Hopper; 1958 - : E.M. Sallee.

## 4. Technical Procedure of Sampling Foods

- Joint FAO/WHO Food Standards Programme

  Code of Principles concerning Milk and Milk Products and Associated Standards (Sixth Edition), FAO/WHO Rome, 1968

  Standard No B-1 (1966)

  Section 3 : Sampling of Milk and Liquid Milk Products(except Evaporated and Sweetened Condensed Milk)

  Section 4 : Sampling of Condensed and Evaporated Milk

  Section 5 : Sampling of Dried Milk and Dried Milk Products

  Section 6 : Sampling of Butter

  Section 7 : Sampling of Cheese.

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APPENDIX III

# METHODS OF ANALYSIS
# AND SAMPLING FOR HONEY

# DRAFT PROVISIONAL STANDARD FOR HONEY

## METHODS OF ANALYSIS AND SAMPLING

## 7.1. DETERMINATION OF REDUCING SUGAR CONTENT

7.1.1 Principle of the method - The method is a modification of the Lane and Eynon (1923) procedure involving the reduction of Soxhlet's modification of Fehling's solution by titration at boiling point against a solution of reducing sugars in honey using methylene blue as an internal indicator. The maximum accuracy for this type of determination is attained by ensuring that the reduction of the Fehling's solution during the standardization step and in the determination of the reducing sugars in the honey solution are carried out at constant volume. A preliminary titration is, therefore, essential to determine the volume of water to be added before the determinations are carried out to satisfy this requirement.

### 7.1.2 Reagents

#### 7.1.2.1 Soxhlet's modification of Fehling's solution

Solution A: Dissolve 69.28g copper sulphate pentahydrate ($CuSO_4 \cdot 5H_2O$; MW=249.71) with distilled water to 1 litre.
Keep one day before titration

Solution B: Dissolve 346g sodium potassium tartrate ($C_4H_4KNaO_6 \cdot 4H_2O$; MW = 282.23) and 100g sodium hydroxide (NaOH) with distilled water to 1 litre. Filter through prepared asbestos.

#### 7.1.2.2 Standard invert sugar solution (10g/1.aq.)

Weigh accurately 9.5g pure sucrose, add 5 ml hydrochloric acid (ca. 36.5% w/w pure HCl) and dilute with water to about 100 ml, store this acidified solution for several days at room temperature (ca. 7 days at 12° to 15°C, or 3 days at 20° to 25°C), and then dilute to 1 litre. (N.B. Acidified 1.0% invert sugar remains stable for several months). Neutralize a suitable volume of this solution with 1N sodium hydroxide solution (40g/1) immediately before use and dilute to the required concentration (2g/1) for the standardization.

#### 7.1.2.3 Methylene blue solution

Dissolve 2g in distilled water and dilute to 1 litre.

#### 7.1.2.4 Alumina cream

Prepare cold saturated solution of alum ($K_2SO_4Al_2(SO_4)_3 \cdot 24H_2O$) in water.

Add ammonium hydroxide with constant stirring until solution is alkaline to litmus, let precipitate settle and wash by decantation with water until wash-water gives only slight test for sulfate with barium chloride solution. Pour off excess water and store residual cream in stoppered bottle.

## 7.1.3 Sampling

### 7.1.3.1 Liquid or strained honey

If sample is free from granulation, mix thoroughly by stirring or shaking; if granulated, place closed container in water-bath without submerging, and heat 30 min. at 60°C; then if necessary heat at 65°C until liquefied. Occasional shaking is essential. Mix thoroughly and cool rapidly as soon as sample liquefies. Do not heat honey intended for Hydroxymethylfurfural or diastatic determination. If foreign matter, such as wax, sticks, bees, particles of comb, etc., is present, heat sample to 40°C in water-bath and strain through cheesecloth in hot-water-funnel before sampling.

### 7.1.3.2 Comb honey

Cut across top of comb, if sealed, and separate completely from comb by straining through [No.40] sieve. When portions of comb or wax pass through sieve, heat sample as in 1.3.1 and strain through cheesecloth. If honey is granulated in comb, heat until wax is liquefied; stir, cool and remove wax.

## 7.1.4 Procedure

### 7.1.4.1 Preparation of test sample - First Procedure (applicable to honeys which may contain sediment)

(a) Transfer 25g accurately weighed sample from the homogenized honey to 100 ml volumetric flask, add 5 ml alumina cream (7.1.2.4) dilute to volume with water at 20°C and filter.

(b) Dilute 10 ml of this solution to 500 ml with water (diluted honey solution).

OR

### 7.1.4.2 Preparation of test sample - Second Procedure

(a) Weigh accurately a representative quantity of about 2g of the homogeneous honey sample, dissolve in distilled water and dilute to 200 ml in a calibrated flask (honey solution).

(b) Dilute 50 ml of the honey solution to 100 ml using distilled water (diluted honey solution).

## 7.1.4.3 Standardization of the modified Fehling's solution

Standardize the modified Fehling's solution A so that exactly 5 ml (pipette), when mixed with approximately 5 ml of Fehling's solution B, will react completely with 0.050g invert sugar added as 25 ml dilute invert sugar solution (2g/1).

## 7.1.4.4 Preliminary titration

The total volume of the added reactants at the completion of the reduction titration must be 35 ml. This is made up by the addition of a suitable volume of water before the titration commences. Since the compositional criteria of the honey standard specify that there should be more than 60% reducing sugars (calculated as invert sugar) a preliminary titration is necessary to establish the volume of water to be added to a given sample to ensure the reduction is carried out at constant volume. This volume of water to be added is calculated by subtracting the volume of diluted honey solution consumed in the preliminary titration (x ml ) from 25 ml.

Pipette 5 ml Fehling's solution A into a 250-ml Erlenmeyer flask and add approximately 5 ml Fehling's solution B. Add 7 ml distilled water, a little powdered pumice or other suitable antibumping agent, followed by about 15 ml diluted honey solution from a burette. Heat the cold mixture to boiling over a wire gauze, and maintain moderate ebullition for 2 min. Add 1 ml 0.2% aqueous methylene blue solution whilst still boiling and complete the titration within a total boiling time of 3 minutes, by repeated small additions of diluted honey solution until the indicator is decolourized. It is the colour of the supernatant liquid that must be observed. Note the total volume of diluted honey solution used (x ml).

## 7.1.4.5 Determination

Calculate the amount of added water necessary to bring the total volume of the reactants at the completion of the titration to 35 ml by subtracting the preliminary titration (x ml) from 25 ml.

Pipette 5 ml Fehling's solution A into a 250 ml Erlenmeyer flask and add approximately 5 ml Fehling's solution B.

Add (25-x) ml distilled water, a little powdered pumice or other suitable antibumping agent and, from a burette, all but 1.5 ml of the diluted honey solution volume determined in the preliminary titration. Heat the cold mixture to boiling over a wire gauze and maintain moderate ebullition for 2 min. Add 1.0 ml 0.2% methylene blue solution whilst still boiling and complete the titration within a total boiling time of 3 min. by repeated small additions of diluted honey solution until the indicator is decolourized. Note the total volume of diluted honey solution (y ml). Duplicate titrations should agree within 0.1 ml.

### 7.1.5 Expression and calculation of results

$$C = \frac{2000}{W \cdot y}$$

where: C = g invert sugar per 100g honey (%)
W = weight of honey sample taken
and y = volume of diluted honey solution consumed in the determination (ml)

### 7.1.6 Notes on procedure

It is essential to the accuracy and repeatability of the determination that the volume of water necessary to bring the reactant mixture to a total volume of 35 ml be determined for each individual sample; the following table gives typical volumes which may be encountered at the preliminary titration stage for the incremental contents of invert sugar shown, assuming the test sample (7.1.4.1) weighs about 25g or test sample (7.1.4.2) weighs about 2g.

| Invert sugar content % | Volume of distilled water to be added ml |
|---|---|
| 60 | 8.3 |
| 65 | 9.6 |
| 70 | 10.7 |
| 75 | 11.6 |

## 7.2. DETERMINATION OF APPARENT SUCROSE CONTENT

### 7.2.1 Principle of the method

Based on the Walker (1917) inversion method.

### 7.2.2 Reagents

7.2.2.1 Soxhlet modification of Fehling's solution (7.1.2.1)

7.2.2.2 Standard invert sugar solution (7.1.2.2)

7.2.2.3 Hydrochloric acid (6.34 N)

7.2.2.4 Sodium hydroxide solution (5 N aqueous)

7.2.2.5 Methylene blue solution 2g/1 litre (7.1.2.3)

### 7.2.3 Sampling

The honey is prepared for sampling as in 7.1.3

### 7.2.4 Procedure

#### 7.2.4.1 Preparation of test sample

Prepare the honey sample as in 7.1.4.1(a). Dilute 10 ml of this solution to 250 ml with distilled water, honey solution (for sucrose determination) OR prepare the honey solution as in 7.1.4.2(a).

#### 7.2.4.2 Hydrolysis of the test sample

The honey solution (50 ml) is placed in a 100-ml graduated flask, together with 25 ml distilled water; heat the test sample to 65°C over a boiling water-bath. The flask is then removed from the water bath and 10 ml of 6.34 N hydrochloric acid added. The solution is allowed to cool to room temperature and neutralized with 5 N sodium hydroxide, using litmus as indicator, cooled again, and the volume adjusted to 100 ml (diluted honey solution).

#### 7.2.4.3 Titration

As in 7.1.4.4

### 7.2.5 Expression and calculation of results

Apparent sucrose content = (Invert sugar content after inversion minus invert sugar content before inversion) x 0.95

The result is expressed as g apparent sucrose/100g honey

## 7.3. DETERMINATION OF MOISTURE CONTENT

### 7.3.1 Principle of method

Based on the refractometric method of Wedmore (1955)

### 7.3.2 Apparatus

Refractometer

### 7.3.3 Sampling

The honey is prepared for sampling as in 7.1.3

### 7.3.4 Procedure

### Determination of the refractive index

Determine the refractive index of the test sample using a refractometer at a constant temperature near 20°C. Convert the reading to moisture content (% w/w) using the table given below. If the determination is made at a temperature other than 20°C, convert the reading to standard temperature of 20°C, according to the temperature corrections quoted. The method used is to be noted in the test report.

### TABLE FOR THE ESTIMATION OF MOISTURE CONTENT
(Wedmore 1955)

| Refractive Index (20°C) | Moisture Content (%) | Refractive Index (20°C) | Moisture Content (%) | Refractive Index (20°C) | Moisture Content (%) |
|---|---|---|---|---|---|
| 1.5044 | 13.0 | 1.4935 | 17.2 | 1.4830 | 21.4 |
| 1.5038 | 13.2 | 1.4930 | 17.4 | 1.4825 | 21.6 |
| 1.5033 | 13.4 | 1.4925 | 17.6 | 1.4820 | 21.8 |
| 1.5028 | 13.6 | 1.4920 | 17.8 | 1.4815 | 22.0 |
| 1.5023 | 13.8 | 1.4915 | 18.0 | 1.4810 | 22.2 |
| 1.5018 | 14.0 | 1.4910 | 18.2 | 1.4805 | 22.4 |
| 1.5012 | 14.2 | 1.4905 | 18.4 | 1.4800 | 22.6 |
| 1.5007 | 14.4 | 1.4900 | 18.6 | 1.4795 | 22.8 |
| 1.5002 | 14.6 | 1.4895 | 18.8 | 1.4790 | 23.0 |
| 1.4997 | 14.8 | 1.4890 | 19.0 | 1.4785 | 23.2 |
| 1.4992 | 15.0 | 1.4885 | 19.2 | 1.4780 | 23.4 |
| 1.4987 | 15.2 | 1.4880 | 19.4 | 1.4775 | 23.6 |
| 1.4982 | 15.4 | 1.4875 | 19.6 | 1.4770 | 23.8 |
| 1.4976 | 15.6 | 1.4870 | 19.8 | 1.4765 | 24.0 |
| 1.4971 | 15.8 | 1.4865 | 20.0 | 1.4760 | 24.2 |
| 1.4966 | 16.0 | 1.4860 | 20.2 | 1.4755 | 24.4 |
| 1.4961 | 16.2 | 1.4855 | 20.4 | 1.4750 | 24.6 |
| 1.4956 | 16.4 | 1.4850 | 20.6 | 1.4745 | 24.8 |
| 1.4951 | 16.6 | 1.4845 | 20.8 | 1.4740 | 25.0 |
| 1.4946 | 16.8 | 1.4840 | 21.0 | | |
| 1.4940 | 17.0 | 1.4835 | 21.2 | | |

### Temperature corrections

### Refractive Index:

Temperatures above 20°C - Add 0.00023 per °C

Temperatures below 20°C - Subtract 0.00023 per °C

## 7.4. GRAVIMETRIC DETERMINATION OF WATER-INSOLUBLE SOLIDS CONTENT

### 7.4.1 Sampling

The honey is prepared for sampling as in 7.1.3

### 7.4.2 Procedure

### 7.4.2.1 Preparation of test sample

Honey (20g) is weighed to the nearest centigram (10 mg) and dissolved in a suitable quantity of distilled water at 80°C and mixed well.

### 7.4.2.2 Gravimetric determination

The test sample is filtered through a previously dried and weighed fine sintered glass crucible (pore size 15-40 microns) and washed thoroughly with hot water (80°C) until free from sugars (Mohr test). The crucible is dried for one hour at 135°C, cooled and weighed to 0.1 mg.

### 7.4.3 Expression of results

The result is expressed as g water-insoluble solids/100g honey.

## 7.5. DETERMINATION OF MINERAL CONTENT (ASH)

### 7.5.1 Sampling

Honey is prepared for sampling as in 7.1.3

### 7.5.2 Procedure

### 7.5.2.1 Ignition of the honey

Honey (5-10g) is weighed accurately into an ignited and pre-weighed platinum or silica dish and gently heated in a muffle furnace until the sample is black and dry and there is no danger of loss by foaming and overflowing. An infra-red lamp may also be used to char the sample before inserting into the furnace. If necessary, a few drops of olive oil may be added to prevent frothing. The sample is then ignited at 600°C to constant weight. The sample is cooled before weighing.

### 7.5.3 Expression of results

The result is expressed as % ash (m/m).

## 7.6. DETERMINATION OF ACIDITY

### 7.6.1 Sampling

The honey is prepared for sampling as in 7.1.3

### 7.6.2 Reagents

7.6.2.1 Sodium hydroxide 0.1N (carbonate-free)

7.6.2.2 Phenolphthalein indicator 1% (w/v) in ethanol, neutralized.

7.6.2.3 Distilled water made carbon dioxide free by boiling and subsequent cooling.

### 7.6.3 Procedure

#### 7.6.3.1 Preparation of test sample

Honey (10.0g) is weighed out and dissolved in 75 ml distilled water (7.6.2.3).

#### 7.6.3.2 Titration

The test sample is titrated against carbonate-free 0.1N sodium hydroxide solution using 4-5 drops of neutralized phenolphthalein indicator. The end-point colour should persist for 10 sec. For darkly coloured samples, a smaller weight should be taken. As an alternative, a pH meter may be used and the sample titrated to pH 8.3.

### 7.6.4 Expression and calculation of results

The result is expressed as milliequivalents acid/1000g honey and is calculated as follows:

Acidity = 10v
where v = the number of ml 0.1N NaOH used in the neutralization of 10g honey.

## 7.7. DETERMINATION OF DIASTASE ACTIVITY

### 7.7.1 Principle of the method

Based on the method of Schade *et al.* (1958) modified by White *et al.* (1959) and Hadorn (1961).

### 7.7.2 Reagents

#### 7.7.2.1 Iodine stock solution:

Dissolve 8.8g of iodine AR in 30-40 ml water containing 22g potassium iodine AR and dilute to 1 litre with water.

7.7.2.2 Iodine solution 0.0007 N:

Dissolve 20g potassium iodine AR in 30-40 ml water in a 500-ml volumetric flask. Add 5.0 ml iodine stock solution and make up to volume. Make up a fresh solution every second day.

7.7.2.3 Acetate buffer - pH 5.3 (1.59 M):

Dissolve 87g sodium acetate . $3H_2O$ in 400 ml water, add about 10.5 ml glacial acetic acid in a little water and make up to 500 ml. Adjust the pH to 5.3 with sodium acetate or acetic acid as necessary, using a pH meter.

7.7.2.4 Sodium chloride solution 0.5 M:

Dissolve 14.5g sodium chloride AR in boiled-out distilled water and make up to 500 ml. The keeping time is limited by mould growth.

7.7.2.5 Starch solution (*):

Use a starch with a blue value between 0.5 - 0.55 using a 1cm cell, as determined by the method below.

Weigh out that amount of starch which is equivalent to 2.0g anhydrous starch. Mix with 90 ml of water in a 250-ml conical flask. Bring rapidly to the boil, swirling the solution as much as possible, heating over a thick wire gauze preferably with an asbestos centre. Boil gently for 3 min., cover and allow to cool spontaneously to room temperature. Transfer to a 100-ml volumetric flask, place in a water bath at 40°C to attain this temperature and make up to volume at 40°C.

Method for determining blue value of starch

The amount of starch equivalent to 1g anhydrous starch is dissolved by the above method, cooled and 2.5 ml acetate buffer added before making up to 100 ml in a volumetric flask.

To a 100-ml volumetric flask add 75 ml water, 1 ml N hydrochloric acid and 1.5 ml of 0.0007 N iodine solution. Then add 0.5 ml of the starch solution and make up to volume with water. Allow to stand for one hour in the dark and read in 1 cm cells using a spectrophotometer at 660 nm against a blank containing all the ingredients except the starch solution.

Reading on the absorbance scale = Blue value.

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* The use of this reagent was endorsed with the proviso that an international reference centre for providing certain chemical reagents such as standard anhydrous starch would be established in the near future.

### 7.7.3 Apparatus

7.7.3.1 Water-bath at 40 ± 0.2°C.

7.7.3.2 Spectrophotometer to read at 660 nm.

### 7.7.4 Sampling

The honey sample is prepared as in 7.1.3 without any heating.

### 7.7.5 Procedure

#### 7.7.5.1 Preparation of test samples

Honey solution: 10.0g honey is weighed into a 50-ml beaker and 5.0 ml acetate buffer solution is added, together with 20 ml water to dissolve the sample. The sample is completely dissolved by stirring the cold solution. 3.0 ml sodium chloride solution is added to a 50-ml volumetric flask and the dissolved honey sample is transferred to this and the volume adjusted to 50 ml.

N.B. It is essential that the honey should be buffered before coming into contact with sodium chloride.

Standardization of the starch solution

The starch solution is warmed to 40°C and 5 ml pipetted into 10 ml of water at 40°C and mixed well. 1 ml of this solution is pipetted into 10 ml 0.0007 N iodine solution, diluted with 35 ml of water and mixed well. The colour is read at 660 nm against a water blank using a 1 cm cell.

The absorbance should be 0.760 ± 0.020. If necessary the volume of added water is adjusted to obtain the correct absorbance.

#### 7.7.5.2 Absorbance determination

Pipette 10 ml honey solution into 50-ml graduated cylinder and place in 40° ± 0.2°C water bath with flask containing starch solution. After 15 minutes, pipette 5 ml starch solution into the honey solution, mix, and start stop-watch. At 5 minute intervals remove 1 ml aliquots and add to 10.00 ml 0.0007 N iodine solution. Mix and dilute to 50 ml. Determine absorbance at 660 nm in spetrophotometer immediately using 1-cm cell. Continue taking 1 ml aliquots at intervals until absorbance of less than 0.235 is reached.

### 7.7.6 Expression and calculation of results

The absorbance is plotted against time (min) on a rectilinear paper. A straight line is drawn through at least the last three points on the graph to determine the time when the reaction mixture reaches an absorbance of 0.235. Divide 300 by the time in minutes to obtain the diastase number (DN). This number expresses the diastase activity as ml 1% starch solution hydrolysed by the enzyme in 1g of honey in 1 h at 40°C. This diastase number corresponds with the Gothe-scale number.

Diastase activity = DN = ml starch solution (1%)/g honey/h at 40°C.

## 7.8. PHOTOMETRIC DETERMINATION OF HYDROXYMETHYLFURFURAL (H.M.F) CONTENT (*)

### 7.8.1 Principle of the method

Based on the method of Winkler (1955)

### 7.8.2 Reagents

7.8.2.1 Barbituric acid solution: Weigh out 500 mg barbituric acid and transfer to a 100-ml graduated flask using 70 ml water. Place in a hot water bath until dissolved, cool and make up to volume.

7.8.2.2 p-toluidine solution: (**) Weigh out 10.0g p-toluidine AR and dissolve in about 50 ml isopropanol by gentle warming on a water bath. Transfer to a 100-ml graduated flask with isopropanol and add 10 ml glacial acetic acid. Cool and make up to volume with isopropanol. Keep the solution in the dark. Do not use for at least 24 hours.

7.8.2.3 Distilled water (oxygen free)

Nitrogen gas is passed through boiling distilled water. The water is then cooled.

### 7.8.3 Apparatus

7.8.3.1 Spectrophotometer to read at 550 nm.

7.8.3.2 Water bath.

### 7.8.4 Sampling

The honey is prepared as in 1.3 without any heating.

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* Method endorsed with the view of replacing it by a spectrophotometric method sometime in the future.

** Caution: p-toluidine is a substance of known carcinogenic properties.

### 7.8.5 Procedure

#### 7.8.5.1 Preparation of test sample

10g of honey sample is weighed and dissolved without heating in 20 ml oxygen-free distilled water (7.8.2.3). This is transferred to a 50-ml graduated flask and made up to volume (honey solution). The sample should be tested after preparation without delay.

#### 7.8.5.2 Photometric determinations

2.0 ml of honey solution is pipetted into each of two test tubes and 5.0 ml p-toluidine solution is added to each. Into one test tube 1 ml water is pipetted and into the other 1 ml barbituric acid solution and both mixtures are shaken. The one with added water serves as the water blank. The addition of the reagents should be done without pause and should be finished in about 1-2 min.

The extinction of the sample is read against the blank at 550 nm using a 1-cm cell immediately the maximum value is reached.

### 7.8.6 Expression and calculation of results

The method may be calibrated by using a standard solution of hydroxymethylfurfuraldehyde (H.M.F.) standardized by dissolving commercial or laboratory prepared HMF and assaying spectrophotometrically where $\varepsilon = 16{,}830$ (J.H. Turner 1954) at 284 nm; using 0-300 µg standards. An equation is given by which results may be roughly worked out:

$$\text{mg/100g HMF} = \frac{\text{Absorbance}}{\text{Thickness of layer}} \times 19.2$$

Results are expressed as mg HMF/kg honey.

## 7.9. LITERATURE REFERENCES

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APPENDIX IV

# METHODS OF ANALYSIS AND SAMPLING FOR PROCESSED FRUITS AND VEGETABLES

# METHODS OF ANALYSIS AND SAMPLING FOR PROCESSED FRUITS AND VEGETABLES

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# METHODS OF ANALYSIS AND SAMPLING FOR PROCESSED FRUITS AND VEGETABLES

The methods of analysis and sampling described hereunder are international referee methods which have been endorsed by the Codex Committee on Methods of Analysis and Sampling.

## I. METHODS OF ANALYSIS

### A. Drained weight

(a) The following method has been endorsed for:

Canned green and wax beans, peaches, grapefruit, sweet corn, pineapple, asparagus, mushrooms (Regular packs, in vinegar, in wine), green garden peas, fruit cocktail, plums, raspberries, strawberries.

### DETERMINATION OF DRAINED WEIGHT - METHOD I

1. **DEFINITION** Drained weight expresses % solid contents as determined by the procedure described below.

2. **MATERIALS**

2.1 Specifications for circular sieves

2.1.1 If the quantity of the total contents of the container is less than 1.5 kg (3 pounds) use a sieve with a diameter of 20 cm (8 inches).

2.1.2 If the quantity of the total contents of the container is 1.5 kg (3 pounds) or more, use a sieve with a diameter of 30 cm (12 inches).

2.1.3 The meshes of such sieves are made by so weaving wire as to form square openings of 2.8 mm by 2.8 mm.(a), (b)

3. **PROCEDURE**

Weigh full can, open, and pour entire contents on circular sieve for which a tare has been established. Without shifting product, incline sieve so as to facilitate drainage. Drain 2 minutes, weigh either drained solids or free liquid direct, and weigh dry empty can.

4. **CALCULATION AND EXPRESSION OF RESULTS**

From weights thus obtained determine % liquid and % drained weight (solid content).

---

(a) Ref. ISO Recommendation R 565.
(b) Such sieves could be replaced by U.S. sieves with no. 8 Standard screen (size of opening 2.38 mm).

## 5. LITERATURE REFERENCES

5.1 A.O.A.C. (1965), 30.00: Drained weight

5.2 ALINORM 69/23, para. 49 (b)

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(b) The following method has been endorsed for:

Canned tomatoes

## DETERMINATION OF DRAINED WEIGHT - METHOD II

1. **DEFINITION** Drained weight expresses % solid contents as determined by the procedure described below.

## 2. MATERIALS

### 2.1 Specifications for circular sieves

2.1.1 If the quantity of the total contents of the container is less than 1.5 kg (3 pounds) use a sieve with a diameter of 20 cm (8 inches).

2.1.2 If the quantity of the total contents of the container is 1.5 kg (3 pounds) or more, use a sieve with a diameter of 30 cm (12 inches).

2.1.3 The meshes of such sieves are made by so weaving wire as to form square openings of 11.2 mm by 11.2 mm. (a), (b)

## 3. PROCEDURE

3.1 Remove lid from container, but in the case of a container with lid attached by double seam, do not remove or alter the height of the double seam.

3.2 Tilt the opened container so as to distribute the contents over the meshes of a circular sieve which has previously been weighed or for which a tare has been established.

3.3 Without shifting the tomatoes, so incline the sieve as to facilitate drainage of the liquid.

3.4 Allow to drain for two minutes.

3.5 At the end of the two minutes draining period, ascertain the weight of the tomato material while still on the sieve, allowing for the tare (or weight of the sieve).

---

(a) Ref. ISO Recommendation R 565.

(b) Such sieves could be replaced by U.S. sieves 2 mesh (size of opening 11.3 mm).

## 4. CALCULATION AND EXPRESSION OF RESULTS

From weights thus obtained determine % liquid and % drained weight (solid contents)

## 5. LITERATURE REFERENCES

5.1 ALINORM 69/23, para 49

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## B. Washed drained weight

The following method has been endorsed for:

Sauce packs, in oil (for canned mushrooms)

## DETERMINATION OF WASHED DRAINED WEIGHT

## 1. DEFINITION

Washed drained weight expresses % solid contents after washing with hot water, as determined by the procedure described below.

## 2. MATERIALS

### 2.1 Specifications for circular sieves

/Fine mesh U.S. sieve No 50/(a) 20 cm (8 inches) diameter.

## 3. PROCEDURE

3.1 Weigh the unopened can.

3.2 Open the can and wash the contents on to a tared fine mesh sieve.

3.3 Wash the contents of the sieve under the running cold water and then wash with running hot water until free of soluble substances.

3.4 Spread the mushrooms after washing over the bottom of the sieve and drain for 5 minutes and then weigh.

3.5 Weigh the empty dried can and determine the net contents (or total product weight).

## 4. CALCULATION AND EXPRESSION OF RESULTS

Calculate the % drained weight on the net contents (or total product weight).

---

(a) To be replaced by the corresponding ISO sieve.

## C. Alcohol insoluble solids

The following method has been endorsed for:

Canned green peas

## DETERMINATION OF ALCOHOL INSOLUBLE SOLIDS

### 1. MATERIALS

#### 1.1 Specifications for circular sieves

1.1.1 If the quantity of the total contents of the container is less than 1.5 kg (3 pounds) use a sieve with a diameter of 20 cm (8 inches).

1.1.2 If the quantity of the total contents of the container is 1.5 kg (3 pounds) or more, use a sieve with a diameter of 30 cm (12 inches).

1.1.3 The meshes of such sieves are made by so weaving wire as to form square openings of 2.8 mm by 2.8 mm. (a), (b)

### 2. PROCEDURE

Pour the sample on circular sieve. Spread peas evenly and let drain. Transfer peas to white pan and remove any foreign material. Add volume $H_2O$ equal to double volume original sample.

Pour peas back on sieve, spreading evenly, tilt sieve as much as possible without shifting peas, and drain 2 minutes. With cloth wipe surplus moisture from lower surface of sieve. Grind drained peas in food chopper until cotyledons are reduced to smooth homogeneous paste, stir, and weigh 20 g ground material into 600 ml beaker. Add 300 ml 80% (v/v) alcohol, stir, cover beaker, and bring to boil. Simmer slowly 30 minutes.

Fit into Büchner filter paper of appropriate size (previously prepared by drying in flat-bottom dish 2 hours at temperature of boiling $H_2O$, covering with tightfit cover, cooling in desiccator, and weighing at once). Apply suction and transfer contents of beaker to Büchner so as to avoid running over edge of paper. Suck dry and wash material on filter with 80 (v/v) alcohol until washings are clear and colourless.

Transfer paper and alcohol-insoluble solids to dish used in preparation of paper, dry uncovered 2 hours at temperature of boiling $H_2O$, place cover on dish, cool in desiccator and weigh at once. From this weight deduct weight dish, cover, and paper.

---

(a) Ref. ISO Recommendation R 565.
(b) Such sieves could be replaced by U.S. sieves with no.8 Standard screen (size of opening 2.38 mm).

3. CALCULATION AND EXPRESSION OF RESULTS

Calculate % by weight of alcohol-insoluble solids.

4. LITERATURE REFERENCES

4.1 A.O.A.C. (1965) 30.015 - Alcohol Insoluble Solids in Canned Peas (6). Official.

4.2 ALINORM 69/23, para 50.

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## D. Total soluble solids

The following method has been endorsed for:

Canned applesauce

### DETERMINATION OF TOTAL SOLUBLE SOLIDS

1. PROCEDURE

Determination by means of refractometer, as described in A.O.A.C. (1965), 29011, 43.009, 43.008.

2. CALCULATION AND EXPRESSION OF RESULTS

Degrees Brix or % by weight of sucrose, without corrections for insoluble solids or acidity, but with corrections for temperatures to the equivalent at 20°C.

3. LITERATURE REFERENCES

3.1 A.O.A.C. (1965), 29011: (Solids) by Means of Refractometer (4). Official, Final action 43.009 : (References tables) Corrections for determining % sucrose in sugar solutions by means of either Abbé or immersion refractometer when readings are made at temperatures other than 20°C, (International Temperature Correction Table, 1936) - 43.008 : Refractive indices of sucrose solutions at 20°C (International Scale, 1936).

3.2 ALINORM 69/23, para. 51.

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## E. Syrup measurements

The following method has been endorsed for:

Canned grapefruit, pineapple, peaches,
fruit cocktail, plums, raspberries,
strawberries.

## SYRUP MEASUREMENTS
(Refractometric method)

### 1. PROCEDURE

Determination by means of refractometer as described in A.O.A.C. (1965), 29011, 43.009, 43.008.

### 2. CALCULATION AND EXPRESSION OF RESULTS

Degrees Brix or % by weight of sucrose, with corrections for temperatures to the equivalent at 20°C.

### 3. LITERATURE REFERENCES

3.1 A.O.A.C. (1965) 29011 : (Solids) by Means of Refractometer (4). Official, Final action - 43.009 :(Reference tables). Corrections for determining % sucrose in sugar solutions by means of either Abbé or immersion refractometer when readings are made at temperatures other than 20°C (International Temperature (Correction Table, 1936) - 43.008 : Refractive indices of sucrose solution at 20°C (International Scale, 1936).

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## F. Test for calcium salts

The following method has been endorsed for:

Canned tomatoes

## DETERMINATION OF CALCIUM IN CANNED VEGETABLES

### 1. PRINCIPLE OF THE METHOD - Complexometric titration

### 2. REAGENTS

Use $H_2O$ redistilled from glass (preferable) or deionized $H_2O$, and the following reagents:

2.1 Potassium hydroxide - potassium cyanide solution

Dissolve 280 g KOH and 66 g KCN in 1 l $H_2O$

2.2 Calcium carbonate - Primary standard grade, dried 2 hours at 285°C.

2.3 Hydroxynaphthol blue - Calcium indicator.(a) Store in dark. Use fresh supply of this indicator after 1 year.

2.4 Ascorbic Acid (AR)

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(a) Mallinckrodt No. 5630 in dispenser bottle ready for use, or equivalent.

2.5 Disodium dihydrogen ethylenediamine tetraacetate (EDTA) standard solution 0.01M. - Dissolve 3.72 g EDTA (more than 99% purity) in $H_2O$ in 1 l volume flask and dilute to volume. Weigh accurately enough $CaCO_3$ to give approximately 40 ml titration with 0.01M EDTA and transfer to 400 ml beaker. Add 50 ml $H_2O$ and enough 10% HCl to dissolve $CaCO_3$. Dilute to approximately 150 ml with $H_2O$ and add 15 ml 1N NaOH, disregarding any precipitate or turbidity. Add approximately 200 mg hydroxynaphthol blue indicator and titrate from pink to deep blue end point, using magnetic stirrer. Add last few ml EDTA solution dropwise.

Molarity EDTA solution = mg $CaCO_3$/(ml EDTA x 100.09).

## 3. APPARATUS

3.1 Titration standard - Fluorescent illuminated, such as Titra-Lite Precision Scientific Co., or equivalent.

3.2 Ion exchange column - Approximately 20 x 600 mm, fitted with coarse porosity sintered glass disk and Teflon stopcock. Place 30-40 g moist Amberlite IR-4B resin (anion exchange resin with high phosphate capacity) in 600 ml beaker and exhaust with three 250 ml portions 5% $Na_2CO_3$ or NaOH. Wash with $H_2O$ until excess base is removed. Treat resin with three 250 ml portions 3% HCl (3 + 22) (a), mixing thoroughly after each treatment. Rinse with $H_2O$ until colour is removed and transfer with $H_2O$ to column. Column is ready for use after draining $H_2O$ to top of resin column. (Exchange capacity for phosphate is approximately 1500 mg; therefore number of aliquots can be passed through column before regeneration is necessary. Rinse column with approximately 250 ml $H_2O$ before each use until eluate is colourless).

## 4. PREPARATION OF SAMPLE

4.1 Liquid from canned whole tomatoes - Drain liquid from tomatoes, centrifuge and pass through fast paper. Weigh 100 g filtrate into Pt or porcelain dish. Evaporate to dryness, using forced-draft oven, infrared radiation, or other convenient means. Ash at temperature not $>$ 525° until apparently C-free (grey to brown). Cool, add 20 ml $H_2O$, stir with stirring rod, and add 10 ml HCl cautiously under watch glass. Rinse off watch glass into dish and evaporate to dryness on steam bath. Add 50 ml HCl (1 + 9) (b), heat on steam bath 15 min., and filter through paper for quantitative analysis into 200 ml volume flask. Wash paper and dish thoroughly with hot $H_2O$. Cool filtrate, dilute to mark, and mix.

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(a) 3 volumes concentrated HCl mixed with 22 volumes of $H_2O$.

(b) 1 volume concentrated HCl mixed with 9 volumes of $H_2O$.

4.2 Canned vegetables - Thoroughly comminute entire contents of can (representative portion if larger than 8 cm diameter can) in high speed blendor. Weigh 50 g sample (100 g sample if there is no declaration of added Ca) into Pt or porcelain dish. Evaporate to dryness using forced-draft oven, irradiation or other convenient means. Ash and treat as for liquid from canned whole tomatoes.

## 5. DETERMINATION

Transfer 50 ml or 100 ml aliquot prepared sample to 250 ml beaker and adjust to pH 3.5 with 10% KOH solution (added dropwise), using pH meter and magnetic stirrer. Pass sample through resin column (column is in chloride form), collecting effluent in 400 ml beaker and adjusting flow rate to 2-3 ml/min. Wash column thoroughly with 100 ml $H_2O$ in two 50 ml portions. Pass first 50 ml through column at the same rate as samples. Pass second portion through at 6-7 ml/min. Finally, pass enough $H_2O$ freely through column to make a total volume 250-300 ml. Mix thoroughly and adjust to pH 12.5-13.0 (using pH meter and magnetic stirrer) with KOH-KCN solution 2.1 (approximately 10 ml). Add 0.100 g ascorbic acid and 200-300 mg hydroxynaphthol blue indicator. Titrate immediately with 0.01M EDTA solution through pink to deep blue end point, using magnetic stirrer.

## 6. EXPRESSION OF RESULTS

50 ml aliquot %Ca = Titration x 0.4008 x 4 x 100/mg sample.
100 ml aliquot %Ca = Titration x 0.4008 x 2 x 100/mg sample.

## 7. LITERATURE REFERENCES

J.A.O.A.C. 1966, 49, 211; 1968, 51, 494.
ALINORM 69/23, para. 53.

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## G. Tough String Test

The following method has been endorsed for:

Canned green beans and canned wax beans.

## TOUGH STRING TEST

## 1. DEFINITION

A tough string is a string that will support the weight of 250 g for five seconds or longer when tested in accordance with the procedure described below.

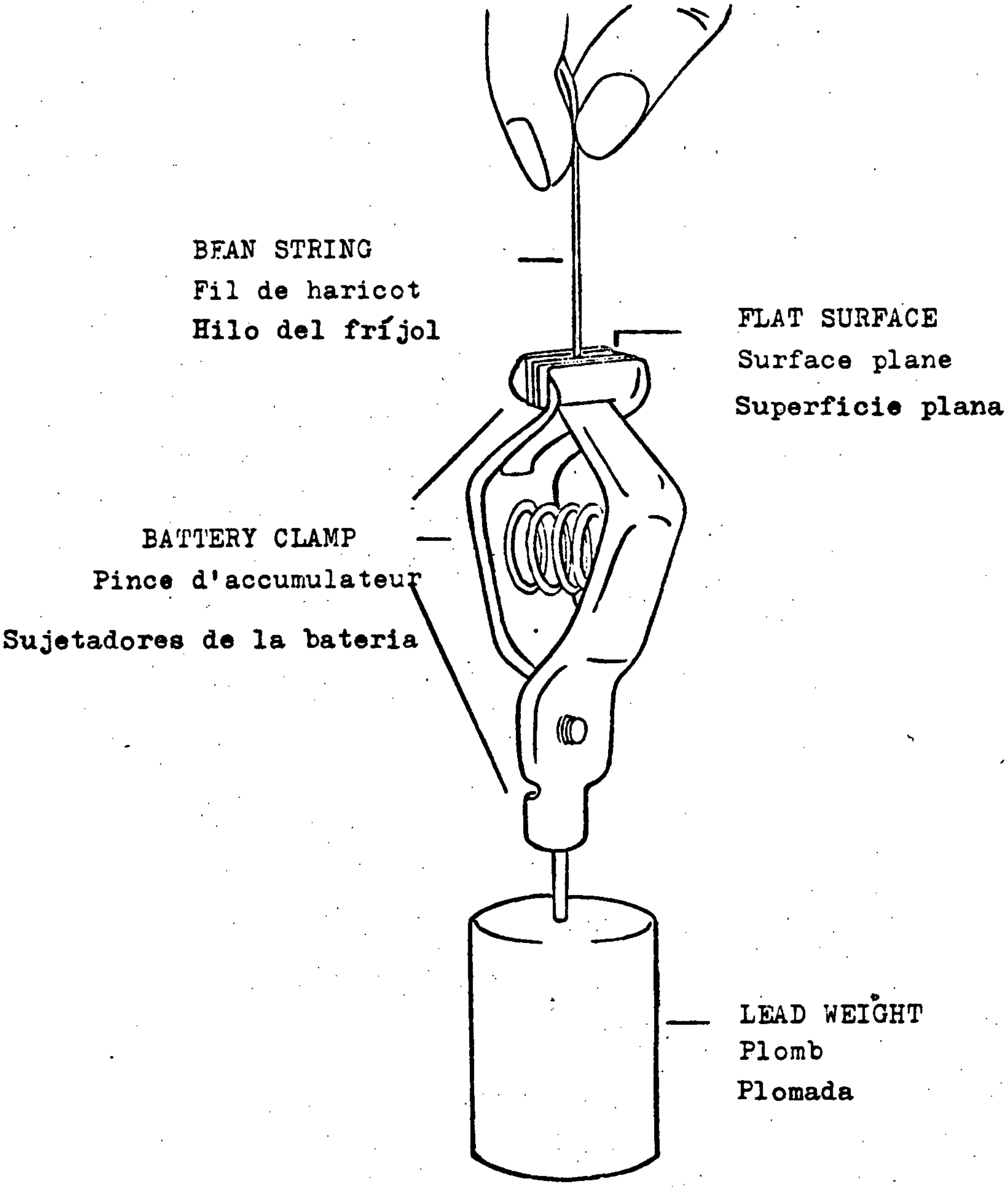
## 2. PRINCIPLE

Strings are removed from individual pods, fastened through a clamp assembly weighing 250 g, and hung so that the string supports the entire weight. If the string supports the weight for five seconds or more it is considered a tough string.

## TOUGH STRING TESTER FOR GREEN OR WAX BEANS

## DISPOSITIF D'EVALUATION DES FILS DURS DES HARICOTS VERTS OU HARICOTS BEURRE

## DISPOSITIVO DE EVALUACION DE LOS HILOS DUROS DE FRIJOLES VERDES Y FRIJOLILLOS



## 3. APPARATUS

### 3.1 Weighted clamp

Use battery clamp (with teeth filed off or turned back), spring operated clothes pin, or binder clip which presents a flat clamping surface. Attach weight so that entire assembly of weight and clamp weighs 250 g. See figure. (A bag containing lead pellets is also convenient as a weight).

## 4. PROCEDURE

4.1 From the drained product select a representative sample of not less than 285 g. Record the weight of this test sample.

4.2 Break the individual bean units and set aside those that show evidence of tough strings. Remove the strings from the pods and retain the pod material for weighing.

4.3 Fasten the clamp assembly to one end of the string. Grasp the other end of the string with the fingers (a cloth may be used to aid in holding the string) and lift gently.

4.4 If the string supports the 250 g assembly for at least five seconds consider the bean unit as containing tough strings. If the string breaks in less than five seconds, retest the broken parts that are 13 mm or longer to determine if such portions are tough.

4.5 Weigh the bean units which contain tough strings and determine % by weight as related to the weight of the test sample, 4.1.

## 5. EXPRESSION OF RESULTS

% by weight of pods containing tough strings equals weight of pods containing tough strings divided by weight of test sample.

## 6. TEST REPORT

Report % pods showing tough strings to nearest 0.1%.

## 7. LITERATURE REFERENCES

ALINORM 69/23, para. 52.

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## H. Mould count method

The following method has been endorsed for:

Canned tomatoes

## MOULD COUNT METHOD

### 1. PRINCIPLE OF THE METHOD

Identification and counting of mould filaments under microscopical examination of a given volume of product, according to Howard's procedure.

### 2. APPARATUS

Howard mould-counting apparatus and accessories for miscroscope (See A.O.A.C. (1965) 36.001).

### 3. SAMPLES

The liquid portion obtained in the "Determination of drained weight, method II", para. 3.

### 4. PROCEDURE

As described in A.O.A.C. (1965) 36.069.

### 5. CALCULATION AND EXPRESSION OF RESULTS

Calculate proportion of positive fields from results of examination of all observed fields and report as % fields containing mould filaments.

### 6. LITERATURE REFERENCES

A.O.A.C. (1965) 36.069 : Molds (12) - Official, Final Action, 36.001, 36.003 (h) (11)

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## I. Method for distinguishing type of peas

The following method has been endorsed for:

Canned green garden peas

## METHOD FOR DISTINGUISHING TYPE OF PEAS

### 1. REAGENTS AND MATERIALS

1.1 Compound microscope - 100 to 250 magnifications
- Phase contrast

1.2 Microscope slide and cover glass

1.3 Spatula

1.4 Ethanol - 95% (v/v)

1.5 Glycerin

## 2. PROCEDURE

### 2.1 Preparing mount

2.1.1 Remove a small portion of the endosperm and place on glass slide;

2.1.2 Using a spatula grind the material with 95% (v/v) ethanol;

2.1.3 Add a drop of glycerin, place cover glass on material and examine under microscope.

### 2.2 Identification

Starch granules of the wrinkled-seeded types (garden peas, sweet) show up as clear cut, well defined, generally spherical particles.

Starch granules of the smooth-seeded types (round, Earlys, Continental) show up as an amorphous mass with no well defined geometric shape.

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## J. Ascertaining proportions of fruit

The following method has been endorsed for:

Canned fruit cocktail

## ACERTAINING PROPORTIONS OF FRUIT

## 1. PROCEDURE

1.1 Determine drained weight and keep liquid and fruit separate;

1.2 Separate individual fruit ingredients, removing those fruits present in lesser amounts (such as cherries, pineapple, grapes);

1.3 Weigh the individual fruit ingredients to the nearest gram;

1.4 Record each fruit's weight and add all of these weights.

## 2. CALCULATION AND EXPRESSION OF RESULTS

Calculate the percentage of fruit proportions:

(a) $\frac{\text{each fruit's weight}}{\text{sum of all fruit weights}} \times 100 = \%$ of the fruit weight

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## II. METHODS OF SAMPLING

### A. Proposed sampling plans for the quality assessment of processed fruits and vegetables

The following method has been endorsed for:

Processed fruits and vegetables

(not applicable to lots at retail level and to factors which might present a hazard to the health of the consumer).

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(a) Do not use the original drained weight of the product before separation of the fruits.

## SAMPLING PLANS FOR PROCESSED FRUITS AND VEGETABLES

This method is described in extenso in ALINORM 69/27 (See also the present report para. 95).

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B. Size of sample unit

(a) The following method has been endorsed for:

Canned pineapple

### SIZE OF SAMPLE UNIT - METHOD I

SAMPLES:

1. In ascertaining the quality requirements for all styles other than Tidbits, Cubes, Crushed or Chips styles, the entire container shall be the sample unit.

2. In ascertaining the quality requirements for Tidbits, Cubes, Crushed or Chips styles, the sample unit shall be:

   2.1 the entire container when it holds 1.0 litre or less; or

   2.2 600 g of drained fruit (of a representative mixture) when the container holds more than 1.0 litre.

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(b) The following method has been endorsed for:

Canned fruit cocktail

### SIZE OF SAMPLE UNIT - METHOD II

SAMPLES:

1. For ascertaining proportions of fruits and fill of container (including drained weight) the entire container shall be the sample unit.

2. For ascertaining compliance with percentage requirements for Sizes and Shapes of fruits and Defects, the sample unit shall be:

   2.1 the entire container when it holds 1 litre or less: or

   2.2 500 g of drained fruit (of a representative mixture) when the container holds more than 1 litre.

3. For ascertaining compliance with requirements based on total contents, the sample unit shall be:

   3.1 the entire container when it holds 1 litre or less; or

   3.2 850 g of fruit and liquid (of a representative and proportionate mixture) when the container holds more than 1 litre.

ALINORM 69/23

APPENDIX V

# GENERAL PRINCIPLES FOR THE ESTABLISHMENT OF CODEX METHODS OF ANALYSIS

Agreed/Procedural Manual

ALINORM 69/23
Appendix V

# GENERAL PRINCIPLES FOR THE ESTABLISHMENT OF CODEX METHODS OF ANALYSIS

## 1. Definition of Codex Methods of Analysis and Sampling

The methods of analysis and sampling contained in the Codex Alimentarius are international referee methods intended for use in case of disputes. These methods will not preclude the use of existing methods for routine inspection or other control purposes.

Where criteria in Codex Standards are related to certain methods of analysis, these methods will be the referee methods.

If further methods have been proven as being equivalent to these methods they may be adopted as alternative methods.

## 2. CRITERIA for the selection of methods of analysis

(a) Official methods of analysis elaborated by international organizations occupying themselves with a food or group of foods should be preferred.

(b) Preference should be given to methods of analysis the reliability of which (accuracy, precision inter- and intra-laboratory variation) has been statistically established in comparative or collaborative studies in several laboratories. Where available, reference should be made to such studies. Speed and simplicity should be of secondary importance in considering methods of analysis for adoption as Codex methods.

(c) Methods of analysis should be selected with a degree of accuracy and precision commensurate with the limits implied in the actual figure given for the analytical criterian in the standard. Priority should be given to methods involving criteria affecting the health of the consumer and also those for which there is a numerical criterion in the standard.

(d) All proposed methods of analysis must have direct pertinence to the Codex Standard to which they are directed.

(e) Methods of analysis must measure the criterion they are claimed to measure. If methods which truly measure the criterion are not available, the need for the criterion should be reconsidered.

(f) Only such methods of analysis should be selected which can ben carried out in laboratories equipped with usual modern apparatus.

(g) Methods of analysis which are applicable uniformly to various groups of commodities should be given preference over methods which apply only to individual commodities.

3. <u>General considerations</u>

(a) The Codex Committee on Methods of Analysis and Sampling should maintain closest possible relations with all interested organizations working on methods of analysis and sampling.

(b) The Codex Committee on Methods of Analysis and Sampling should organize its work in such a manner as to keep under constant review all methods of analysis and Sampling published in the Codex Alimentarius.

(c) In the Codex methods of analysis, provision should be made for variations in recent concentrations and specifications from country to country.

(d) Codex methods of analysis which have been derived from scientific journals, theses, or publications, either not readily available or available in languages other than the official languages of FAO and WHO, or which for other reasons should be printed in the Codex Alimentarius <u>in extenso</u>, should follow the standard layout for methods of analysis as adopted by the Codex Committee on Methods of Analysis and Sampling.

(e) Methods of analysis which have already been printed as official methods of analysis in other available publications and which are adopted as Codex methods need only be quoted by reference in the Codex Alimentarius.

# STANDARD LAYOUT FOR A CODEX METHOD OF FOOD ANALYSIS

## GENERAL

1. TITLE
2. SCOPE
3. FIELD OF APPLICATION
4. DEFINITIONS
5. PRINCIPLE OF METHOD, REACTIONS, SPECIFICITY
6. REAGENTS AND/OR MATERIALS
7. APPARATUS
8. SAMPLING AND SAMPLES
9. PROCEDURE
10. CALCULATION AND EXPRESSION AND INTERPRETATION OF RESULTS
11. SPECIAL CASES
12. NOTES ON PROCEDURE
13. TEST REPORT
14. SCHEMATIC REPRESENTATION OF PROCEDURE
15. LITERATURE REFERENCES
16. APPENDICES

## Notes on the Use of the Standard Layout

The Notes propose the headings to the used and include general directions for the drafting of methods of food analysis. Though designed primarily to cover chemical, physical and organoleptic tests they are applicable also, with suitable amendments, to microbiological tests.

## I. GENERAL

1. Application of the layout - In making use of this standard layout, it must be remembered that it is for use as a general layout for guidance only, and should be adapted to suit any particular requirements. There may be no need in some cases for all the sub-divisions provided, and these should then be ignored. Within this limitation, the adoption of this standard form of layout and drafting will ensure :

- that the various items of information to be included in the standard are set out always in the same order ;
- that no important point is overlooked in the preparation of the standard ;
- that the section sought may be found rapidly, whatever the origin or scope of the standard.

(This is important, in particular when dealing with a partial translation of a text, or when comparing two texts).

2. Plan of document - In drafting the method, the subjects should be dealt with in the order shown in the standard layout, omitting however any section, clause or heading, which may be unnecessary in the particular case in question, and adding, if required, in the most appropriate place, any further ones to cover specialized subjects not already provided for.

If the document relates solely to methods of analysis, items such as 'Characterization of product', 'Requirements', which have a proper place only in the specifications for the food product itself, should not be included.

3. Numbering - A single consecutive decimal point numbering system should be used throughout, for the sections and clauses of each document, also where the document contains several parts, each covering a different method of test.

4. Units and symbols -

4.1 The units, signs and symbols laid down in ISO Recommendations* or failing these, those already well known internationally, should be used whenever possible.

The millilitre (ml) is usually used as the special name for the cubic centimetre ($cm^3$), in accordance with the decision of the XII th General Conference of Weights and Measures (Paris, October 1964).

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* See in particular ISO Recommendation R 31.

The symbols for units of measurement should be used, instead of the terms themselves, whenever they are preceded by a number expressed in figures ; but in other cases, these units should generally be written out in full (a typical exception, is in the column heading in tables of numerical values, where it is more usual to show the units in the form of their symbols only).

4.2 "%" Sign Unless for aesthetic reasons to the contrary, or to meet particular typographical requirements (e.g. in titles etc.), the % sign should be used in the place of the words "per cent", in English, "pour cent" in French and "Prozent" in German, in the sections "Reagents" and "Expression of results", and elsewhere, in the Codex document, when it is preceded by figures. (Example : write "5% (m/m) solution" but "five per cent copper content").

5. Terminology in general

5.1 When, in one Codex language, there is a choice of different terms available to express a given concept, preference should be given to that which resembles most the equivalent term or terms in the other Codex languages (e.g. End point = point final = Endpunkt).

5.2 When there is no similarity of terms in the different languages to express the same concept, and when there exist internationally recognized symbols or abbreviations which can be employed, these should be used in parenthesis after the terms themselves in the different languages, in order to assist readers of all languages.

6. Choice and wording of methods of test - As far as possible and except where, in the field in question, this would be contrary to a reasonable and well established practice which it is desirable to retain, the same methods should be adopted in all Codex documents for the determination of a given characteristic in the same product or in related food products, and their wording should be as similar as possible. If the method selected is already given in another Codex document, or in an ISO Recommendation, and it is not thought necessary to repeat it again, the phrase 'use the method described in document ... or 'use one of the methods described in document (s)....' should be used, if necessary with an indication of any modifications.

7. Chemical nomenclature - the principles of chemical nomenclature laid down by the International Union of Pure and Applied Chemistry, including its rules for the spelling and printing of names and symbols, should be followed. Where there is a common name, this may with advantage be used in parentheses in certain cases after the IUPAC name.

8. Terminology of solutions

8.1 Aqueous solutions.

Solutions for which the solvent is not specified, are, by convention, aqueous solutions ; the use of the term 'aqueous', in defining these solutions, is therefore optional and in general superfluous.

## 8.2 Reagent solutions

8.2.1 Equivalent terms - the following equivalent terms in English, French, Spanish and German, should be used to describe reagents solutions :

| EQUIVALENT TERMS | | | |
|---|---|---|---|
| English | French | Spanish | German |
| standard volumetric solution | solution titrée | solución valorada | Normal-lösung |
| standard reference solution | solution étalon de référence | solución patrón de referencia | Kontroll-lösung |
| standard solution | solution étalon | solución patrón | Standard-lösung |
| standard matching solution (*) | solution témoin | solución testigo | Standard vergleichs-lösung |

(*) This English term is used solely as a generic term for this type of solution and each solution should be defined more precisely to show the characteristic to be matched, e.g. 'Standard colorimetric solution', 'Standard turbidimetric solution', etc.

These terms are defined as follows :

8.2.1.1 Standard volumetric solution. A solution used in titrations, the strength of which may either be known in advance of its use in the test, or which is applied in the titrations during the test itself, with or without its strength having actually to be determined as a part of the procedure.

8.2.1.2 Standard reference solution. A solution used as a reference solution for standardizing other solutions. These solutions are prepared either from a primary standard or standardized by some other means.

8.2.1.3 Standard solution. A solution of accurately known concentration in terms of a particular element, ion, compound or group, derived from the product used for its preparation. Example : standard ferric solution, standard sulphate solution.

8.2.1.4 Standard matching solutions. Solutions of which the relevant characteristic named is exactly known or defined (for example colour, turbidity, etc.) and is used in a matching test to assess the strength of the test solution through the medium of that characteristic. They may be prepared from solutions 8.2.1.1, 8.2.1.2, 8.2.1.3 or other solutions with the required characteristic.

## 8.2.2 Expression of strength of solutions

8.2.2.1 Standard volumetric solutions. The strength of these solutions should be expressed in terms of normality or molarity, the latter being used only when this is necessary to avoid any ambiguity. The strength is represented by a whole number (N, 2N, M, etc.) or by a decimal number (0.1 N, 0.06 N, 2.5 N, 0.5 M, etc.). In certain cases, the strength of the solution may be given in grammes per litre, and the abbreviation g/l should then be used when it is preceded by a numerical value expressed in figures.

8.2.2.2 Standard reference solutions. The strength of these solutions should be expressed in the same way as that of standard volumetric solutions (8.2.2.1).

8.2.2.3 Standard solutions. The strength of these solutions should usually be expressed in grammes per litre or in sub-multiples thereof.

8.2.2.4 Standard matching solutions. The strength of these solutions should be expressed in accordance with the indications of clauses 8.2.2.1, 8.2.2.2 and 8.2.2.3, as applicable.

8.2.2.5 Other solutions.

8.2.2.5.1 Where the concentration of a solution is to be given on a mass by mass or volume by volume basis, its value should be calculated as a percentage, and written in the following way :

... % (m/m) or ... % (v/v)

8.2.2.5.2 In cases where the concentration of the solution is expressed in terms of the dimensionally heterogeneous units of mass and volume, the value should be expressed in grammes per litre or in sub-multiples thereof, where appropriate.

8.2.2.5.3 In cases where a solution is prepared by dilution of another specified solution, the following conventions should be observed :

"diluted $p_1 \rightarrow p_2$" means that $p_1$ parts by volume of the specified solution are diluted to give $p_2$ parts by volume of final mixture.

"diluted $p_1 + p_2$" means that $p_1$ parts by volume of the specified solution are added to $p_2$ parts by volume of the solvent.

<u>NOTE</u> (i) Expressions such as "$p_1 : p_2$" or "$p_1/p_2$" which have different meanings in different countries, should not be used.

<u>NOTE</u> (ii) Traditional or commonly used expressions of strength other than those described in the above sub-clauses (8.2.2.1 to 8.2.2.5), e.g. "hydrogen peroxide, 12 volumes", should not be used.

9. <u>Density and relative density.</u>

- <u>Density</u> (symbol $\rho$) is the quotient of the mass by the volume. The unit of density to be adopted is the gramme per cubic centimetre ($g/cm^3$) for which the symbol g/ml may be used.

- <u>Relative density</u> (symbol <u>d</u>) is the ratio of the density of a homogeneous body to the density of a reference body under conditions which should be specified for both bodies.

10. <u>Terminology of samples</u>

- The "Laboratory sample" is the product as prepared for submission to the laboratory.

- The "Test sample" is the product in the state of preparation in which it is subjected to the test.

- The "Test portion" is the quantity of product actually drawn from the test sample (or, if both are the same, from the laboratory sample ) and on which the test is actually carried out.

| EQUIVALENT TERMS | | | |
|---|---|---|---|
| English | French | Spanish | German |
| Laboratory sample | Echantillon pour laboratoire | Muestra de laboratorio | Laboratoriumsprobe |
| Test sample | Echantillon pour essai | Muestra de ensayo | Testprobe |
| Test portion | Prise d'essai | Porción para ensayo | Testportion |

## II NOTES ON THE INDIVIDUAL SUBJECT SECTIONS

### 1. TITLE

The title should reflect lucidly and concisely the contents of the standard, indicating the nature of the determination and the products to which it applies. If it is to be in several parts, these should go from the general to the particular.

Example : "Milk. Determination of fat content. Gravimetric reference method, based on Röse-Gottlieb".

### 2. SCOPE

The scope should define the contents of the standard, i.e. the method of test and the products to which it applies. It should contain in particular any additional, but useful, information which is too extensive to be included in the title. In simple cases, it may define also the applicability of the method; this avoids then the need for a separate subject heading "Field of application".

### 3. FIELD OF APPLICATION

This section should contain all information on the field of application of the standard to enable the user to judge quickly whether the standard is applicable to the products being considered, or whether limitations exist.

It should contain in particular :

- an indication of the products to which the method of analysis applies and, if there are several methods in the standard, a means of differentiating clearly their respective fields of application;

- the limits between which the method can be used without alteration for the determination of a given characteristic or group of characteristics (e.g. element) in a given product or group of products; these limits take into account the presence of the other constituent characteristics or elements of the product or products in question, and their own limiting contents.

A clear distinction should be drawn between the individual fields of application of each of the methods laid down, whenewer the standard includes several methods, as is indeed sometimes necessary for the determination of a given component, depending for example on the nature of the products to be tested, on their differing contents of that component, or on the accuracy of results required.

11. <u>Use of text notes</u>. These are intended for emphasis or to give helpful and additional information which is not necessarily an essential part of the method. They should be kept to a minimum and only used when it is not convenient to give the information in an Appendix. They should be placed at the end of the paragraph to which they refer, and should be given a number if it is desired to refer to them in connection with a specific word or phrase in the text.

12. <u>Footnotes</u>. These are intended only for reference and for information regarding sources of materials. <u>They should never carry any information or instructions essential to the method</u>.

If it is necessary to provide for modifications to the basic method, for instance to eliminate interferences due to the presence or absence of certain components or because the product is in a different form, these modifications should be treated as "special cases" and dealt with as such in section 11, reference thereto being included in the present section.

Information on the usefulness of the method for assessing product quality, and on any statutory regulations, can also be included.

## 4. DEFINITIONS

If definitions or descriptions of certain terms are necessary to make the text more readily understood, these should be given in this section.

## 5. PRINCIPLE OF METHOD, REACTIONS, SPECIFICITY

The "principle of method" clause should indicate briefly the essential steps of the method used, if necessary giving the reasons justifying the choice of certain procedures; it should preferably use substantive phrases (e.g. "titration with ...") rather than the corresponding narrative, imperative or infinitive forms.

In the case of chemical methods, and where this is considered useful for a better comprehension of the text or of the basis of the calculations, it is recommended that a brief indication of the reactions involved be given - but only for guidance and not claiming thereby to settle any controversial questions. Such indications may help in particular to understand the method, when, in the determination for instance of a given element, several successive changes in the valency of that element occur during the procedure.

If particulars concerning the specificity of the method are required, they may also be given either in this section or in Section 13 "Notes on procedure".

Additional information should be given either in a note to this section or in Section 13.

## 6. REAGENTS AND/OR MATERIALS

This section lists all the reagents, solvents, indicators, and other materials, required during the test, excluding however those required only in the preparation of other reagents. It should give their essential characteristics and, particularly in the case of reagents, their composition, degree of purity and concentration, together with, if necessary, the precautions to be taken for their storage and the time for which they may be stored.

When special standard solutions are to be used, their preparation and, if required, their standardization, should be described, or, if this has been the subject of an ISO Recommendation, a reference to that Recommendation should be given. When the absence of an interfering element in the reagents has to be verified, it may be useful also to give the details of the tests to be used for this purpose.

The list should be drawn up in a systematic order, grouping the items, for example in the following sequence :

i) Pure products (excluding solutions),

ii) Solutions or suspensions of approximate strength stated (excluding standard solutions),

iii) Standard solutions of exact strength stated,

iv) Indicators.

Each item should be identified by a number; by showing this number in parentheses, in the text of the procedure next to its short name, it is possible to avoid having to repeat all the characteristics of the item in question and thus to keep the text short.

In the list, use chemical names or recognized common names only, and avoid trade names and names of patented products, unless a specific product of this type is required for a well defined reason. In this case show the trade name if possible only in a footnote incorporating the phrase"..... has been found satisfactory for this purpose".

For each item, give first the chemical name or common name, followed by descriptive terms, for instance :

(1) Potassium hydroxide, solid sticks.

(2) Sodium hydroxide, standard solution, 0.1 N.

(3) Starch, solution 1% (m/m).

When instructions are given for the preparation and standardisation, these should follow immediately, making the sentences as concise as possible.

6.1 Quality of reagents, etc. It is desirable to provide above the list of reagents, etc., a statement on the general characteristics, and special characteristics to which the materials should conform.

In particular, reagent grade chemicals should be specified where necessary, and the quality of the water or other solvents to be used clearly indicated.

6.2 Pure products. Pure products should be described unambiguously, giving all the particulars necessary for their clear identification, e.g. the recognized chemical name and/or common name by which they are usually known, and by their chemical formula if without it the description might be ambiguous, (including, for solid products in particular, their water of crystallisation if any).

6.3 Expression of strength of solutions - The strength of the solutions listed should be expressed in the manner described in Section I.8 above.

6.4 Correction factors in relation to solution strengths - For standard volumetric solutions given in the list of reagents, the strength indicated in terms of round figures should not be assumed to be necessarily exactly that which must be employed, as the analyst can often be allowed some latitude in this respect. Where such latitude is used, an appropriate correction factor will have to be applied in the method and in particular in the final calculation of the results of the test.

In this case, it is recommended that no reference to such correction factors should be made in the "Expression of results" clause as such, but that the following standard note, or one like it, should appear in an appropriate place in the document, and that attention be drawn to it in the "Reagents" and "Expression of results" clauses.

Note : If the standard solutions or standard volumetric solutions used are not of exactly the strentgh indicated in the list of reagents, a suitable correction factor should be employed in calculating the results and elsewhere, where appropriate, in the procedure.

## 7. APPARATUS

This section should list in a logical order the apparatus required for the test, excluding common laboratory apparatus but including specially modified forms or unusual sizes of such equipment. In addition, apparatus which, though commonly available in laboratories, requires a description too long to be conveniently included in the procedure clauses, and apparatus which plays a significant part in the safety, precision or reproducibility, of the method should be included. Where appropriate, reference should be made to any existing ISO Recommendations, and special types of apparatus and their assembly might usefully be illustrated by a diagram drawn, in this case, in accordance with relevant ISO Recommendations.

Apparatus used only in the preparation of the reagents should not normally be included.

This section should also include any instructions necessary for the cleaning, assembly, adjustment or standardization, of apparatus to be used, where this is not more properly included under "Procedure".

## 8. SAMPLING AND SAMPLES

8.1 Laboratory sample (see definition in Section I.10). The sampling necessary for, and prior to, the preparation of the laboratory sample, is not generally considered a part of the method of analysis as such, and it is usually sufficient in this section of the document to refer to the relevant standard dealing specifically with this question, or to the corresponding section of the product standard.

Where no such texts exist, however, it may be necessary to include a suitable sampling plan and sampling procedure, in this section, drawn up taking into account the recommendations on terminology and statistical treatment in this field, laid down by ISO/TC 69 "Statistical treatment of a series of observations".

This procedure should then specify the operations necessary to obtain a laboratory sample truly representative of the material (or lot) on which the analysis is to be carried out; and can usefully include information on the required volume or mass of the sample, and on the means of preserving the latter, e.g. characteristics of the sample container (type, capacity, air-tightness, etc.) and storage conditions to be used.

8.2 Preparation of the test sample (see definition in Section I.10). This clause should give all the necessary information for the preparation of the "test sample" from which, for the purpose of the test in question, the "test portions" will be drawn. This sample is prepared direct from the "laboratory sample" (Clause 8.1) or may be derived indirectly from it as the result of operations in connexion with another test. In the latter case, the origin of the test sample should be clearly indicated e.g. "Solution A (or filtrate B, or precipitate C) from the determination of...". In either case, all the steps in the preparation should be stated, (e.g. evaporation, grinding, sieving, drying, dissolving, etc.), together with appropriate information on the size of sample required, type of container, and storage conditions advocated, and on any other relevant characteristic (e.g. pH).

## 9. PROCEDURE

This section should describe in detail the successive steps of the procedure, and be divided for this purpose into as many clauses as there are separate steps or sequences of operations to be carried out. Each of these operations should be described accurately, preferably in the chronological order of execution, grouping the steps into clauses and paragraphs in a logical way to facilitate the description, comprehension and application of the method.

If the numbers of steps is large, it is recommended that the clauses covering each be divided into further clauses each corresponding to a given analytical operation.

The following clauses are those usually included under "Procedure" ; any that are not relevant are omitted however and others added, as required, in each particular case.

9.1 Apparatus. Give under this heading any detailed instructions for the cleaning, assembly, adjustment and standardization of the apparatus, that may be necessary or unique to the method, and which, if they are not more appropriately included in section 7 above, can properly be treated as part of the "Procedure". Specify in particular any operating details necessary, before the test, to ensure that the apparatus is functioning correctly.

9.2 Reference standards and calibration curves. Give detailed instructions for the preparation of calibration curves or tables and on the use of reference standards, including standard samples used to verify the validity of the method or to assure uniformity of test technique.

9.3 Safety precautions. If there are risks of explosion, fire, toxicity or other hazards, and special precautions are necessary, prominent cautionary statements should be included in the procedure, in capitals or bold italics, at the appropriate place in the text. More detailed advice on safe procedure, and first aid hints, can then be left to an Appendix.

9.4 Test portions. This clause should give all the information necessary for the drawing of the test portions from the test sample (Clause 8.2) (or from the laboratory sample if both are the same). Where appropriate, it should state in particular the method of weighing or of measuring these test portions (e.g. using a weighing pipette). It should state the mass or volume of the test portions, the precision with which this has to be measured, and any other relevant characteristics.

9.4.1 Measuring out test portions by mass. There are usually two ways of measuring out test portions by mass :

(a) the operator is required to weigh out, with the specified precision, an exactly prescribed quantity of test sample,

and (b) the operator is required to take an approximately stated quantity of test sample and then, by weighing it with the precision specified to ascertain its exact mass.

These cases call for different forms of wording, as in the following examples :

Example of case (a) : "weigh to the nearest 0.001g, exactly 5g of test sample"

Example of case (b) : "weigh to the nearest 0.001g, approximately 5g of test sample".

> Note. In this context, the expression "approximately" is generally assumed to mean "within ± 10%" of the figure stated. Should a smaller tolerance be envisaged, it is recommended that this be clearly indicated, e.g. (in the above example) :
>
> "weigh to the nearest 0.001 g, 4.8 to 5.2 g of test sample".

9.4.2 Measuring out test portions by volume. There are two recommended forms of words for indicating the precision to which test portions are to be measured out ; these are

(a) when the precision is to be implied from that of the apparatus used : by stating what the latter is, for instance : "measure out 10 ml of the test sample with a pipette precise to 0.005 ml"

(b) when the precision of the apparatus to be used is not stated : by indicating in the conventional way the numerical value of the volume in question to the precision required, for instance "measure out 10.00 ml of test sample" or (this being the same) "measure out 10 ± 0.005 ml of test sample".

9.5 Blank test. A blank test for the control of the method as a whole, or of part of it, or of the purity of the reagents used, should be specified whenever necessary, and the conditions for carrying out this test should be clearly stated under this heading. The blank test should normally be performed in parallel with the analysis itself, using the same procedure as in the latter, and the same quantities of all reagents, but omitting the product being tested. In certain cases, however, this absence, in the blank test, of the components of which the product is made, may cause the conditions in the blank test to differ so much from those in the test itself that these differences interfere with the application of the method. In such cases, the modifications in the procedure which must be applied in the blank test or in an auxiliary blank test, to rectify these differences while retaining the same quantities of reagents as in the test itself, should be clearly laid down in this clause.

9.6 Determination(s). The operations of which the determination is made up should be described preferably in their chronological order of execution, and set out in an easily readable form to assist in understanding and applying the procedure. Should it be necessary, in the course of these operations, to keep the product of one of these steps in the procedure (e.g. filtrate or precipitate) for use as a "test sample" in a later determination, this should be clearly stated and provision made for it to be given a reference symbol or letter by which it can be identified again later. In this case, a form of words such as the following could usefully be employed in the relevant clauses of the methods :

- "keep filtrate (D) for the determination of ...."

and later : - "use filtrate (D) from the determination of ...."

## 10. CALCULATION, EXPRESSION AND INTERPRETATION OF RESULTS

This section should give instructions on how the results are to be calculated and assessed.

10.1 Methods of calculation and formulae. This clause is used only when the test provides numerical values of observations, and requires results obtained by calculation from these values. It should then give all the instructions necessary for this calculation of the results, including the instructions for any corrections to be applied to the observations made during the test. For this purpose, this clause should give the complete formula for the calculation in terms of letter symbols, accompanied, where appropriate, by a simplified formula; it should define the meaning of these symbols; including the units in which they are expressed, and it should state unambiguously the item measured, e.g. by its full chemical formula.

To achieve uniformity, the following letter symbols are recommended :

Mo or Vo, for the mass or volume of the test portion

$M$, $M_1$, $M_2$, etc., for values of mass

$V$, $V_1$, $V_2$, etc., for values of volume

An example or presentation of this clause is as follows :

"The percentage, by mass, of arsenic oxide, $As_2O_3$, in the product is equal to :

$$0\ldots\ldots \times V \quad \frac{1000}{500} \quad \times \frac{100}{M_o}$$

where

$M_o$ is the mass, in grammes of the test portion,

$V$ is the volume, in millilitres, of solution A (see Section 9)

Take as the result the arithmetic mean of the determinations, if the conditions of repeatability have been satisfied. Give the result to four significant places.
If the method requires the use of standard solutions, the following note should be included :

"If the standard solutions or standard volumetric solutions used are not of exactly the strength indicated in the list of reagents, a suitable correction factor should be used in calculating the results".

10.2 Significant figures and rounding off rules. Experimental data and results of calculations thereon should be to a number of significant figures logically related to the sensitivity and precision of the method taking also into account how truly representative the test sample was of the products under test. Rounding off should be employed as the last operation in calculation, and conform to the following rules :

"If the first digit discarded is greater than 5, or if it is a 5 followed by at least one figure other than 0, increase the last digit retained by one unit.

If the first digit discarded is a 5 followed by zeros, round upward the last digit retained if it is an odd number; make no adjustment if it is an even number.

If the first digit discarded is less than 5, make no adjustment to the last digit retained.

## 10.3 Precision and reliability.

10.3.1 Sensitivity of method. This should be borne in mind, and referred to where it is a factor in the significance of the observed values and therefore in the interpretation of the results.

10.3.2 Precision of method. This is defined as the closeness of agreement between the results obtained by applying the experimental procedure several times under the prescribed conditions. Combined with a knowledge of any possibility of bias in the method, it is a measure of the accuracy and reliability of the method. When comparative tests have been carried out on the method, it is recommended that the precision be indicated under the two headings of repeatability and reproducibility, which are defined and should be expressed as follows :

- Repeatability. The closeness of agreement between replicate results obtained with the same method, on identical test material, by the same analyst, in the same laboratory, using the same apparatus, at the same time or in quick succession. It should be expressed in the text of the method as follows :

  "The difference between the results of two determinations carried out simultaneously or in rapid succession by the same analyst should bot exceed....."

- Reproducibility. The closeness of agreement between individual results obtained with the same method on identical test material but under different conditions (different analysts, different apparatus, different laboratories and/or different times). It should be expressed in the text of the method as follows :

  "The difference between the results obtained in two different laboratories on the same sample should not exceed....."

## 10.4 Possibility of bias.

Any possibility of a systematic error or bias in the method as such, or in the results of the observations made, should be discussed briefly, and assessed numerically if possible, especially if it is likely to account for differences between the results obtained using alternative standard methods for the determination in question.

## 10.5 Overall (absolute) assessment of results.

An overall interpretation of the results of the analysis should finally be required under this heading, on the basis of recognized statistical principles and taking into account the various factors referred to above, which serve to judge the acceptability of these results.

## 11. SPECIAL CASES

This section brings together all the alterations in the procedure necessitated by the presence or the absence of specific components in the product to be analysed, or because the product is in a different form. The alterations required on this account should already have been referred to in Section 3, "Field of application". Each special case should be given an exact sub-title.

The text of the modified method should include the following sub-divisions :

- Principle, i.e. the alteration or alterations in the priciple of the general procedure, or, possibly, the principle of the new procedure ;
- Sampling, i.e. any alteration in the general method of sampling ;
- Procedure, i.e. the new procedure or a statement of the alterations envisaged. If a partial modification is in question, it is necessary to relate the altered part clearly to the general procedure by indicating the last paragraph of the unaltered procedure and its position in the text, or preferably by completely reproducing it. The altered part is then set out, and the first unaltered paragraph which follows the alteration is referred to in the same way.
- Expression of results, i.e. that applicable to the modified or additional procedure.

It is preferable, even if the alteration is very slight, to draft a section entitled "Special case", rather than describe this alteration in a note, since the above procedure is preferable from the point of view of clarity.

## 12. NOTES ON PROCEDURE

Any notes intended to complete the description of the procedure and to facilitate the application of the method, should be placed at the end of the paragraph or clause to which they refer. However, if these necessitate a relatively long text, they should be placed under a special section 'Notes on procedure' placed after "Procedure".

## 13. TEST REPORT

This section should give all the indications necessary for the identification of the sample, the method used, the results and the form in which they are expressed and any operations not specified in the standard or regarded as optional, which might affect the result.

## 14. SCHEMATIC REPRESENTATION OF PROCEDURE

Particularly with long procedure consisting of multiple operations, it is sometimes useful to have a schematic representation of the different stages of the procedure. Such a diagram should be drawn so as to separate clearly the different steps and to group on a vertical line the sequence of operations carried out at a given intermediate state (precipitate, filtrate, residue, etc.). It should state explicitly if a given sequence of operations ends at a given stage (e.g. "filtrate to be thrown away).

## 15. LITERATURE REFERENCES

This section will be provided only in those cases where the number of literature references is large or where there is merit in grouping them together under a separate heading. In other cases, it may be more convenient to show them in the text itself of the other sections concerned, or in the footnotes thereto. Only references to publications supporting, or providing needed supplementary information, should be given. References that are merely historical, or for acknowledgment, are not desirable.

## 16. APPENDICES

Appendices should be used when it is desirable to relieve the body of the document of detailed information which can more conveniently be presented in that form. Examples of this are :

- Valuable, but not strictly essential, information which has not been incorporated in the text, with a view to simplification.
- A description of the merely recommended forms of apparatus.
- Full dimensional drawings, with detailed description, of apparatus, where required.
- A variant of the method, given as an example only.
- An alternative method which may be used for routine purposes, but which it is not desired to include in the main text of the standard.
- Tables for the calculation of results.
- A numerical example of the calculations or test report.
- Literature extracts.
- Experimental results on which the precision of the method has been determined.
- Recommended safe procedure and first aid hints.

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APPENDIX VIII

# RELATIVE DENSITY

# OF

# EDIBLE FATS AND OILS

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Appendix VIII

# RELATIVE DENSITY OF EDIBLE FATS AND OILS

The following method for the determination of relative density has been endorsed for: Edible oils (except olive oil), lard and rendered pork fat, premier jus and edible tallow:

## DETERMINATION OF THE RELATIVE DENSITY AT 20°C OF FATS AND OILS

### 1. SCOPE

This method is applicable to fats and oils which are liquid and which do not deposit stearin at the temperature of determination.

### 2. DEFINITION

The relative density at $t/20^\circ$C of an oil or fat is the ratio of the weight in air of a given volume of the oil or fat at $t^\circ$C to the same volume of water at 20°C, the weighings being made with weights adjusted to balance brass weights in air.

### 3. PROCEDURE

Calibrate a relative density bottle or pycnometer (of capacity at least 25 ml) as follows. Clean and dry the bottle and weigh it; fill it with freshly boiled and cooled distilled water and keep it in a bath of water at 20°C until it reaches that temperature. If a bottle is used, insert the stopper in such a way that the capillary is completely filled with water, and then maintain it at 20°C until no further alteration in volume occurs. Wipe the stopper. If a pycnometer is used, adjust the volume of liquid to the fixed mark. Remove the bottle or pycnometer from the bath, dry the outside, allow to stand for a short time and weigh.

Empty and dry the bottle or pycnometer. Fill it with the sample of oil previously brought near to the temperature of $t^\circ$C. Keep the bottle or pycnometer in a bath adjusted to $t^\circ$C until it has acquired that temperature. If a bottle is used, insert the stopper in such a way that the capillary is completely filled with the oil and then maintain it at the temperature $t^\circ$C until no further alteration in volume occurs. Wipe the stopper. If a pycnometer is used, adjust the volume to the fixed mark. Remove the apparatus from the bath, dry the outside, allow to stand for a short time and weigh. Make all weighings in air with weights adjusted to balance brass weights in air.

### 4. CALCULATION AND EXPRESSION OF RESULTS

$$\text{Relative density at } t/20^\circ\text{C in air} = \frac{W_2}{W_1 \left[ 1 + \alpha(t - 20^\circ) \right]}$$

where $W_2$ = weight in grammes of oil obtained in the test

$W_1$ = weight in grammes of water obtained in calibration test

and $\alpha$ = the coefficient of cubic expansion of glass at the given temperature

= 0.000 03 for soda glass

= 0.000 01 for borosilicate glass

## 5. NOTE

Provided that no stearin can separate from the oil at temperature in the neighbourhood of t°C, and that no visible moisture or impurities are present in the oil, the relative density may be determined at any temperature between (t + 5)°C and (t - 5)°C. The relative density at t°C is calculated from the figure so obtained by adding 0.000 69 to the figure determined for each Centigrade degree by which the temperature of observation exceeds t°C or by subtracting from it 0.000 69 for each Centigrade degree by which it is lower than t°C.

## 6. LITERATURE REFERENCES

6.1 British Standard 684:1958, p.10; Determination of the specific gravity at t/15.5°C in air and the apparent density (g/ml) in air of fatty oils. Method 1.

(REPRODUCED FROM REPORT OF THIRD SESSION
OF CODEX COMMITTEE ON METHODS OF ANALYSIS
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Appendix IX

# PROPOSED METHODS OF ANALYSIS AND SAMPLING FOR SUGARS

## I. METHODS OF ANALYSIS

### 1. WHITE SUGAR

| Analytical Criterion | Method | Accuracy [a] | Reference |
|---|---|---|---|
| Polarization expressed as sucrose | ICUMSA method for raw sugars (without lead). Lead defecation only if necessary; no correction for 'lead effect' | ± 0.1°S | Ic.R1958p.84 |
| Invert Sugar | KNIGHT and ALLEN method for contents below 0.02% | ± 0.005% | Ic.M. p. 29 |
| | BERLIN INSTITUTE method for contents between 0.02% and 0.10% | ± 0.005% | Ic.M. p. 25 |
| | LANE and EYNON method for contents above 0.1% | ± 0.01% | Ic.M. p. 13 |
| Conductivity Ash | ICUMSA method for conductivity ash using 5g/100 ml or 28g/100g solutions; with 5g/100ml, the conductivity expressed in micromhos/cm should be multiplied by the standard C-ratio factor of $18 \times 10^{-4}$ | ± 0.001% | Ic.R1962 p.12 |
| Loss on drying | ICUMSA method using a minimum sample size of 20g (without grinding) | ± 0.005% | Ic.M.p.44 |
| Colour (ICUMSA units) | Measurement by ICUMSA method 4 on a solution of 50g/100g after filtration through a membrane filter of pore size 0.4 μ to 0.6 μ. Results to be expressed as 'ICUMSA units' as defined in Ic.M.p.58 | not yet established | IcM.pp 57 and 58<br>IcR 1958 p.52 |
| Sulphur Dioxide | CARRUTHERS, HEANEY and OLDFIELD method. The range and accuracy depend on the concentration of the test solution | | |
| | 1-7 mg/kg test solution of 40g/100ml | ± 0.3 mg/kg | |
| | 2-15 mg/kg test solution of 20g/100ml | ± 0.5 mg/kg | |

## 1. WHITE SUGAR (Continued)

| Analytical Criterion | Method | Accuracy a/ | Reference |
|---|---|---|---|
| Sulphur Dioxide (continued) | 5-30mg/kg, test solution of 10g/100ml | ± 1.0mg/kg | I.S.J.1965 p.364 |
| | 10-60mg/kg, test solution of 5g/100ml | ± 2.0mg/kg | |
| Arsenic (As) | Diethyldithiocarbamate method using wet ashing and colorimetric measurement with silver diethyldithiocarbamate | | AOAC 1965 24.011 |
| Lead (Pb) | ICUMSA method with wet ashing satisfactory below a level of 0.5mg/kg | ± 0.1mg/kg | Ic.M. p.48(c) |
| Copper (Cu) | ICUMSA method with wet ashing for levels referred to in Codex standards | ± 0.5mg/kg | Ic.M. p.106 |

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a/ See paragraph 8(d) of this report

## 2. SOFT SUGARS

### (a) White to Dark Brown

| Analytical Criterion | Method | Accuracy a/ | Reference |
|---|---|---|---|
| Sucrose (saccharose) + invert sugar expressed as sucrose | TATE and LYLE Invertase Modification of LANE and EYNON method | | Ic.M.p.71 |
| Invert Sugar | LANE and EYNON method (without inversion) | | Ic.M.p.71 |
| Sulphated Ash | Gravimetric double sulphation method | | Ic.M.p.36 |
| Loss on drying | ICUMSA method using 10g | | Ic.M.p.44 |
| Sulphur Dioxide | CARRUTHERS, HEANEY and OLDFIELD method<br>The range and accuracy depend on the concentration of the test solution | | |
| | 1-7mg/kg, test solution of 40g/100ml | ± 0.3mg/kg | |
| | 2-15mg/kg, test solution of 20g/100ml | ± 0.5mg/kg | |
| | 5-30mg/kg, test solution of 10g/100ml | ± 1.0mg/kg | I.S.J.1965 p.364 |
| | 10-60mg/kg, test solution of 5g/100ml | ± 2.0mg/kg | |
| Arsenic (As) | Diethyldithiocarbamate method using wet ashing and colorimetric measurement with silver diethyldithiocarbamate | | AOAC, 196 24.011 |
| Lead (Pb) | ICUMSA method with wet ashing, satisfactory below a level of 0.5mg/kg | ± 0.1mg/kg | Ic.M.p.48(c) |
| Copper (Cu) | ICUMSA method with wet ashing for levels referred to in Codex standards | ± 0.5mg/kg | Ic.M.p.106 |

2. SOFT SUGARS (Continued)

(b) Soft White Sugar

| Analytical Criterion | Method | Accuracy [a] | Reference |
|---|---|---|---|
| Sucrose (saccharose) + invert sugar expressed as sucrose | TATE and LYLE Invertase modification of LANE and EYNON method | | Ic.M.p.71 |
| Conductivity Ash | ICUMSA method for conductivity ash using 5g/100ml or 28g/100g solutions; with 5g/100ml the conductivity expressed in micromhos/cm should be multiplied by the standard C-ratio factor of $18 \times 10^{-4}$ | | Ic.R.1962p.12 |
| Colour (ICUMSA units) | Measurement by ICUMSA method 4 on a solution of 50g/100g after filtration through a membrane filter of pore size 0.4/u to 0.6/u | | Ic.M.pp.57 and 58<br>Ic.R.1958 p.52 |

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[a] See paragraph 8(d) of this report

## 3. LACTOSE

| Analytical Criterion | Method | Accuracy [a] | Reference |
|---|---|---|---|
| Lactose anhydrous | LANE and EYNON method | | Ic.M.p.13 |
| Sulphated Ash | Single sulphation | | Ic.M.p.100 |
| Loss on drying | U.S.P.method (drying 16 hours at 130°C.) or KARL FISCHER method | | U.S.P. 1965 p.336<br>Angew.Chem. 1935,48,394 |
| pH(10% solution) | by pH meter | | Ic.M.p.44 |
| Arsenic (As) | Diethyldithiocarbamate method using wet ashing and colorimetric measurement with silver diethyldithiocarbamate | | AOAC,1965 24.011 |
| Lead (Pb) | ICUMSA method with wet ashing satisfactory below a level of 0.5mg/kg | ± 0.1mg/kg | Ic.M.p.48(c) |
| Copper (Cu) | ICUMSA method with wet ashing for levels referred to in Codex standards | ± 0.5mg/kg | Ic.M.p.106 |

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[a] See paragraph 8(d) of this report

## 4. GLUCOSE SYRUP, DRIED GLUCOSE SYRUP

| Analytical Criterion | Method | Accuracy a/ | Reference |
|---|---|---|---|
| Total solids | Drying in vacuum oven | | C.I.R.F.Method E.42 |
| Dextrose Equivalent (reducing sugars expressed as D-glucose | LANE and EYNON method | | Ic.M.p.101 |
| Sulphated ash | Single sulphation method | | Ic.M.p.100 |
| Sulphur dioxide | CARRUTHERS, HEANEY and OLDFIELD method. The range and accuracy depend on the concentration of the test solution, namely | | |
| | 1-7 mg/kg test solution of 40g/100ml | ± 0.3 mg/kg | |
| | 2-15mg/kg test solution of 20g/100ml | ± 0.5 mg/kg | |
| | 5-30mg/kg test solution of 10g/100ml | ± 1.0 mg/kg | |
| | 10-60mg/kg test solution of 5g/100ml | ± 2.0 mg/kg | I.S.J.1965 p.364 |
| | -600mg/kg test solution of 0.5g/100ml | ± 20 mg/kg | |
| Arsenic (As) | Diethyldithiocarbamate method using wet ashing and colorimetric measurement with silver diethyldithiocarbamate | | AOAC, 1965 24.011 |
| Lead (Pb) | ICUMSA method with wet ashing satisfactory below a level of 0.5 mg/kg | ± 0.1 mg/kg | Ic.M.p.48(c) |
| Copper (Cu) | ICUMSA method with wet ashing for levels referred to in Codex standards | ± 0.5 mg/kg | Ic.M.p.106 |

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a/ See paragraph 8(d) of this report

## DEXTROSE MONOHYDRATE and DEXTROSE ANHYDROUS

| Analytical Criterion | Method | Accuracy[a] | Reference |
|---|---|---|---|
| Dextrose (expressed as D-glucose) | LANE and EYNON method | | Ic.M p.101 |
| Total solids | Drying at 100°C for 4 hours under reduced pressure | | Ic.M p.113 |
| Sulphated ash | Single sulphation method | | Ic.M p.100 |
| Sulphur dioxide | CARRUTHERS, HEANEY and OLDFIELD method. The range and accuracy depend on the concentration of the test solution, namely: | | |
| | 1-7mg/kg test solution of 40g/100ml | ± 0.3 mg/kg | |
| | 2-15mg/kg test solution of 20g/100ml | ± 0.5 mg/kg | |
| | 5-30mg/kg test solution of 10g/100ml | ± 1.0 mg/kg | |
| | 10-60mg/kg test solution of 5g/100ml | ± 2.0 mg/kg | I.S.J.1965 p.364 |
| Arsenic (As) | Diethyldithiocarbamate method using wet ashing and colorimetric measurement with silver diethyldithiocarbamate | | AOAC 1965 24.011 |
| Lead (Pb) | ICUMSA method with wet ashing, satisfactory below a level of 0.5 mg/kg | ± 0.1 mg/kg | Ic.M p.48(c) |
| Copper (Cu) | ICUMSA method with wet ashing for levels referred to in Codex standards | ± 0.5 mg/kg | Ic.M p.106 |

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[a] See paragraph 8(d) of this report

## II. METHOD OF SAMPLING
(for White and Soft Sugars)

### 1. Sampling from bags (Ic.M. p.86)

Preferably, samples should be taken from opened bags. When this is not possible samples should be taken by piercing the bags with a trier of the type described in Ic.M. p.80, the punctures being sealed by appropriate means, e.g. with adhesive tape for paper bags. For domestic packets up to 5 kg in weight, the entire package should be taken.

### 2. Number of samples to be taken

The maximum size of a lot is 500 tons. The number of packages to be sampled will be $\sqrt[3]{\ }$(where T is the tonnage of the lot) with a minimum of three packages. In all cases, the gross sample set apart will be at least 2 kg: more than one insertion of the trier being made when necessary.

### 3. Preparation of the sample for analysis (Ic.M. p.83)

From the gross sample collected as described above and mixed, 4 sub-samples, each of at least 500g, should be prepared by the ICUMSA method for raw sugars and sealed in moisture-proof containors.

## III. ABBREVIATIONS USED IN THE APPENDIX

Ic.M. = ICUMSA Methods of Sugar Analysis (1964)

Ic.R. = ICUMSA Report (Report of the Proceedings of the Session)

I.S.J. = International Sugar Journal

C.I.R.F. = Corn Industries Research Foundation

A.O.A.C. = Association of Official Analytical Chemists